

Chemical Age

A.E.A.'S NEW
URANIUM
FACTORY
(page 199)

VOL. 81 No. 2064

31 January 1959

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31 January 1959

CHEMICAL AGE

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on high pressure
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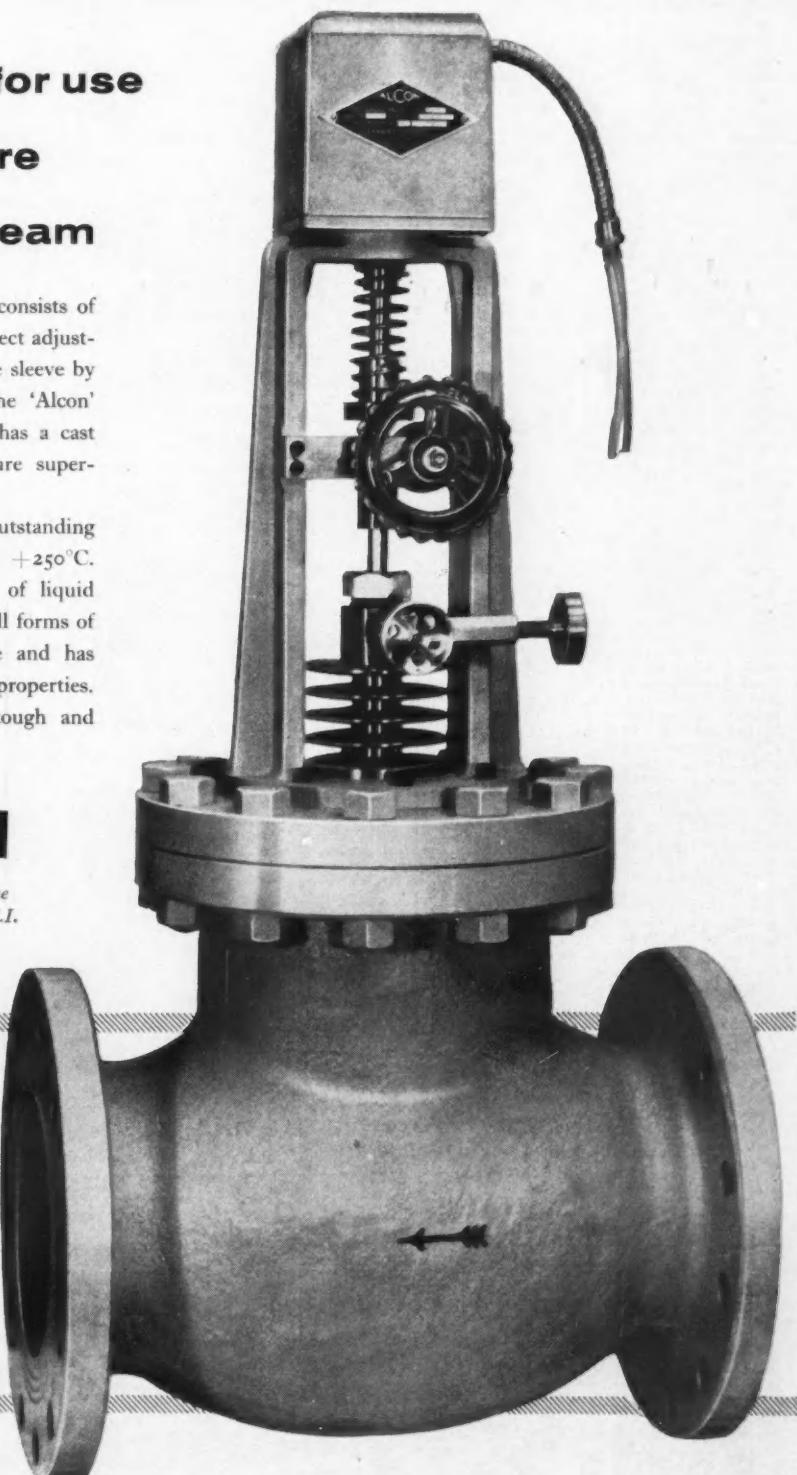
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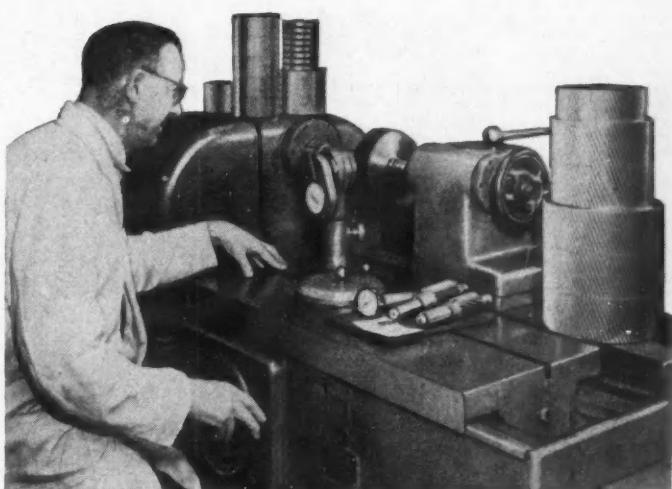


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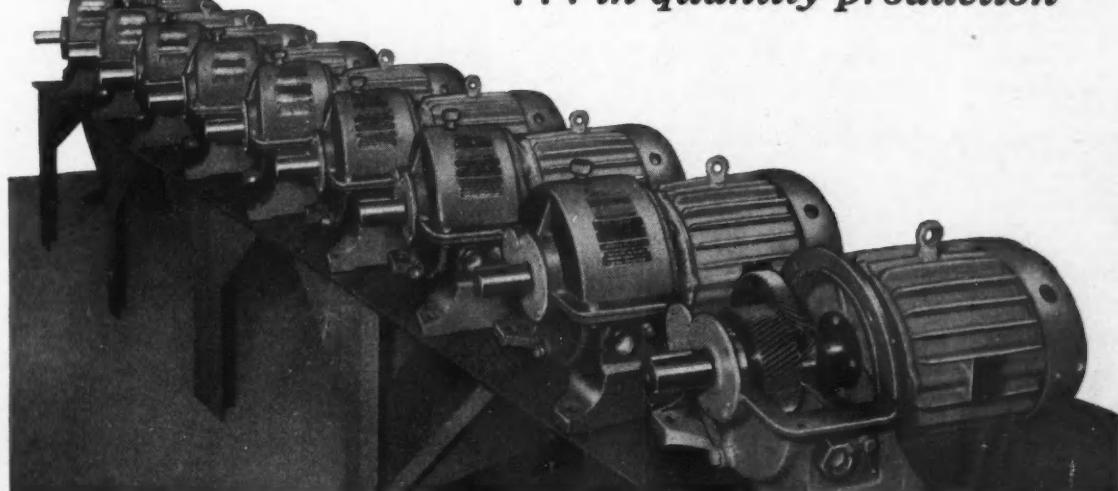
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13 June 1959

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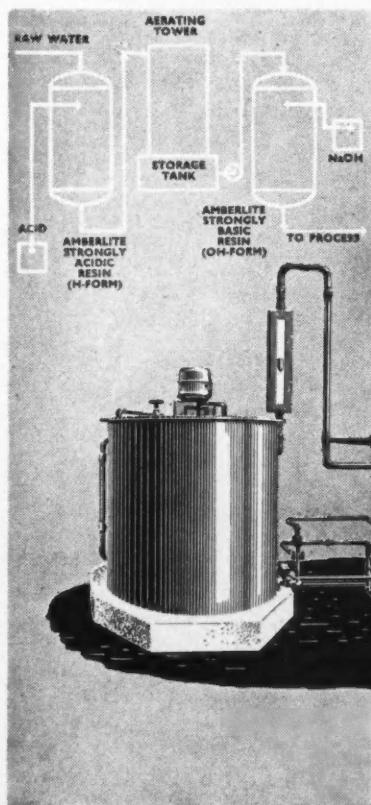


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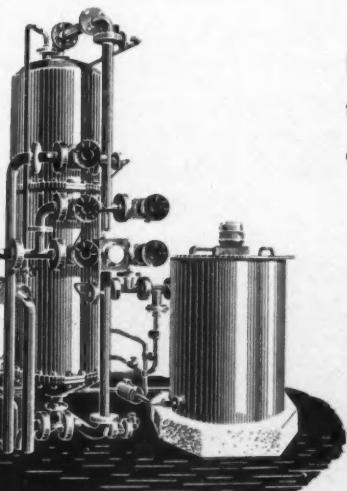


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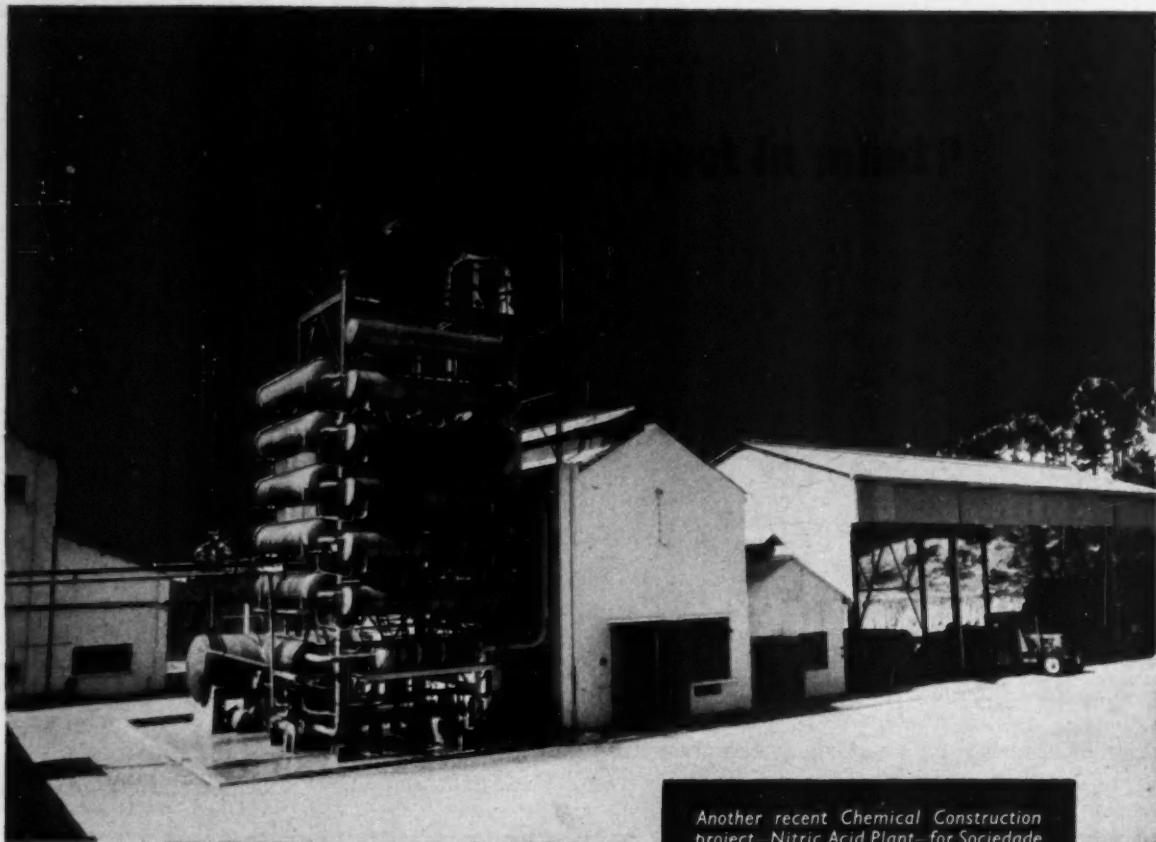


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VOL. 81

No. 2064

JANUARY 31 1959

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CHEMICAL AGE

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CHEMICAL EXPORTS

YEAR-END recovery in U.K. exports of chemicals was not sufficient to make good the slacker period in mid-year, but they accounted for a higher-than-ever proportion of total U.K. exports. Total for the year, at £263,132,000 compared with the highest-ever figure of £267,432,000 in 1957, but was considerably higher than in the previous record year of 1956 when the total was £244,541,000.

In view of the extremely difficult trading conditions, the 1958 export performance of the British chemical industry was highly commendable. Not only did the industry have to contend with minor recessions in some of the major using industries as well as fiscal restrictions, but competition from other major producing countries intensified sharply. The fact that many formerly non-producing countries have been setting up their own chemical companies is also having an effect on our export trade.

The fall in total chemical exports experienced during 1958 was only one of 2% compared with the previous year; the value of all U.K. exports declined by 3½%. Chemicals accounted for their highest-ever proportion of total U.K. exports, the 1958 figure being 8.2%, against 8% in 1957; 7.6% in 1954; 6.8% in 1948; and an average of 6.3% in the period 1935 to 1938.

The classification of chemicals in the Trade and Navigation Accounts includes a number of broad headings and export trade in them during 1958 was as follows: chemical elements and compounds, down by 4% at £60.4 million (against £62.6 million in 1957 and £59.4 million in 1956); pigments, paints, etc., down 2% at £23.7 million (£24.2 million in 1957 and £23.5 million in 1956); drugs and medicines, down 5% at £37.8 million (£39.6 million in 1957 and £35.9 million in 1956); soaps, toilet preparations, etc., down 9% at £26.2 million (£28.7 million in 1957 and £26.8 million in 1956); plastics materials up 8% at £32.2 million (£29.9 million in 1957 and £26.1 million in 1956).

Individually, the export performance for a number of chemicals was remarkably good. For instance, 1958 shipments of organic acids, anhydrides and their salts and esters, valued at £1,712,443, was higher than 1957 by £351,000, while exports of ethyl alcohol, at £1,704,927, rose by £118,000; phenol shipments, at £1,035,447, increased by £154,000. Increases were also registered for inorganic acids, aluminium oxide, chloride of lime, calcium compounds, lead compounds, sodium phosphates, sodium silicate, zinc oxide, acetone, pigment-dyestuffs, ammonium sulphate, etc.

During 1958 the export of British drugs and medicines to the U.S. reached the record figure of £1,100,000, compared with £830,000 in 1957. Exports to Egypt recovered, from £84,000 to £494,000, and the industry believes that the signing of the Anglo-Egyptian financial agreement will lead to considerably more sales in this market. Antibiotics made up the biggest section of this trade (£8.3 million) with vitamins next (£2.5 million). Exports of alkaloids, aspirin and sulphonamide preparations each exceeded £1 million.

Imports of chemicals were 5% higher at £119.8 million compared with £114.3 million in 1957 and £106.9 million in 1956. Imports of drugs and medicines and plastics materials in particular were higher. Imports of p.v.c. were known to have risen—the 1958 figure is given as £3,270,373, but separate figures for imports in 1957 were not provided.

LOWERING HELIUM COSTS

WITH the current importance of helium to national defence in the U.S. and to both basic and applied research, to the fore, attention has been focused on the cost of its transportation. The U.S. National Bureau of Standards' Boulder Laboratories have been studying this problem. The Bureau now reports that liquefaction and transportation of the gas at or near its liquid density will lower costs by reducing shipping charges. Moreover, this procedure would also facilitate distribution.

At present, distribution of the extracted and purified gas from production plants to consumers requires the use of extremely heavy, high-pressure cylinder tank cars having a limited gas capacity. Consequently, transportation costs form an appreciable percentage of the total gas delivered. The number of uses for helium as well as an increased overall demand today has resulted in consumption of helium being 600% greater than it was a decade ago. Chief applications of the gas now are rocketry, shielded arc welding, whether balloons, nuclear reactors, aeronautical research and cryogenic investigations.

The N.B.S. process for helium liquefaction employs work-producing expansion engines and requires the use of liquid nitrogen or liquid air as pre-coolants to produce the necessary refrigeration. This eliminates pre-cooling by liquid hydrogen with its accompanying hazards and operation complexity. Another desirable result is greater cycle efficiency.

The operation suggested is a simple Claude-type cycle which consists of one expansion engine operating at a temperature below that of a liquid nitrogen pre-coolant bath. A modification, made possible by a 155-atmosphere pressure differential, employs a second expansion engine in the helium-feed gas stream for the conservation of the liquid nitrogen refrigerant. Pressure of the helium feed from the production plant is 170 atmos. while the operating pressure of the liquefier is only 15 atmos.

Pipelines insulated and shielded with liquid nitrogen or liquid air make possible the efficient transfer of helium from the liquefier to transportable containers. However, a new insulation has been suggested by private industry which has a thermal conductivity low enough to eliminate the need for liquid nitrogen shielding.

During transportation, isolation of the container allows the pressure to rise to some fixed design value based on travel time and rate of heat of transfer, accomplishing 'no-loss' storage. Adequate for transportation with the continental U.S. are newly designed tank cars with an inner helium vessel capable of withstanding a 10 atmos. pressure rise within a 10- to 15-day period. Stainless steel meets

the requirements for the pressure vessel. Carbon steel or aluminium are the preferred metals for the outer ambient temperature vessel.

Two methods are proposed for the transfer of helium. If an adequate pressure is maintained in the container, helium can be forced through a heat exchanger, warmed to ambient temperature, and then piped to gas storage cylinders through conventional compression equipment. In the second method a low-temperature pump can be used to force the helium through a heat exchanger to compressed gas storage.

Considerable attention has been paid to the thermodynamic problems associated with helium liquefaction and transportation. These are reducing the amount of liquid helium required to cool the storage vessel and eliminating temperature stratification of helium in the container. The 'cool-down' of equipment in which liquid helium is the only refrigerant is wasteful because of helium's low heat of vaporisation. To reduce this waste, N.B.S. has adopted a well-known cryogenic technique so that a portion of the sensible heat of the cold helium gas that has been vaporised during the initial stages of the 'cool-down' could be utilised. This has been accomplished by incorporating a concentric shell inside the helium pressure vessel to channel the evaporated helium between the vessel and pressure shell. It is stated that this decreases the amount of liquid helium required for cooling by a factor of 10. An operational solution to the second problem, however, is not yet available. Early results of an investigation currently under way provide a method of minimising temperature stratification so that it is possible to keep temperature inside the vessel quite uniform so that pressure will not rise more rapidly than predicted. Also being studied is the design of transport containers of an optimum size with respect to minimum gross weight and maximum performance. The liquefied gas, it is reported, will be transported by insulated rail tank car having a capacity of approximately 13,000 gall.

N.B.S. studies have been extended, also, to the design of liquefaction facilities with an hourly rate of 260 gall. Truck-trailers with capacities of 6,000 gall. have been proposed for the transportation of less than car-load quantities. Small quantities of liquid helium are now also air-shipped in the U.S. in 12.5 and 19 gall. helium containers. The only special precaution has been a check valve on the normal vent line to prevent air from being drawn into the liquid helium space by changes in atmospheric pressure. Losses during such shipments have been found to be less than $\frac{1}{2}$ gall.

SOLID BORON HYDRIDE

ACCORDING to Dr. R. Köster of the Max Planck Institute, Mulheim, West Germany, the complete hydrogenation of the liquid boric trialkyl BR_3 , carried out at temperatures of around 200°C, in a complicated reaction process, leads to the formation of solid, non-volatile, mainly yellow-brown coloured boron compounds, containing hydrogen and carbon as well as boron. By carrying out the reaction in the proper manner, the total boron content of the boron alkyl, Dr. Köster claims, may be obtained in concentrated form as a solid material.

By hydrogenating the solid product, it is simple to change the carbon content, Dr. Köster reports, of the yellow-brown powder into volatile hydrocarbons. When this is done, a light yellow to colourless boron hydride with 80% to 90% boron content is produced. The high-

polymer substances, insoluble in organic solvents, resemble, in their properties the yellow (BH) compounds produced from diborane and other hydrogen borides by A. Stock and others (*Berichte*, 1936, **69**, 1469; *J. Amer. Chem. Soc.*, 1933, **55**, 4009).

The nearly colourless substances change under applications of heat to a brown or brown-black product which contains more than 90% elementary boron. The only possible impurities apart from a little carbon are small quantities of oxygen.

It is understood that research in this field is continuing. The work noted above was completed by January 1958, but at Dr. Köster's own request has only just been announced.

URANIUM PROCESSING AT SPRINGFIELDS

Automatic Methods

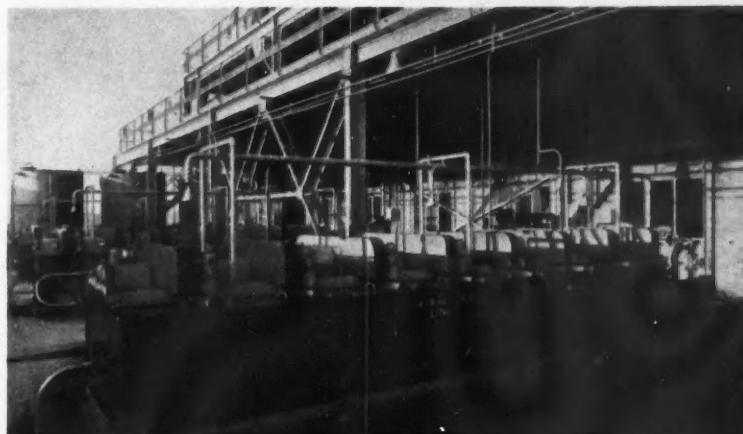
Adopted at A.E.A.'s New Continuous Process Plant

BRITAIN'S atomic energy project depends, at present, entirely on uranium. All the source material, in the form of ore or concentrates, is processed at the U.K. Atomic Energy Authority's Springfield Works, Lancs, to produce pure uranium metal for reactor fuel or uranium hexafluoride for the gaseous diffusion plant at Capenhurst.

The metal is formed into rods and assembled into fuel elements for the Authority's reactors at Calder Hall and Chapelcross, as well as for the BEPO reactor at Harwell. Shortly, fuel elements for the nuclear power stations being built by the industrial consortia for the electricity undertakings both at home and overseas, will also be manufactured at Springfield. (See CHEMICAL AGE, last week, p. 171).

The plant at Springfield has been developed since 1946, when the British atomic energy project was started. Because of the urgency attached to the project, a wartime defence plant which had been producing mustard gas, was acquired and modified. Formidable problems in designing the plant were met. Imperial Chemical Industries Ltd. had developed a process for conversion of uranium oxide from the U.S. to metallic uranium. At the start-up of the plant, for the early stages—the extraction from ore and production of crude oxide—only laboratory scale information was available. Pilot-plant work was out of the question because of the time element and the old plant was designed on laboratory scale findings, and included a new process to replace part of that developed by I.C.I. Batch processes for most of the plant were used to simplify the job.

Since the design organisation was completely new with no files of drawings for reference, every item of the plant had to be designed from scratch. The standard of purity specified for the product made it necessary to consider levels of contamination measured in parts per million. In particular, there was the hazard due to the radioactivity of material to be handled by the ton. At that time, i.e. 1946, there was far less experience on permissible exposures and practically none on the design of plant most effective in protecting workers. The change from pitchblende to concentrate as the starting material has eliminated, however,



Fully automatic solvent extraction process in which uranyl nitrate is removed from other dissolved impurities

the gamma radiation hazard. Today, workers in the new plant wear for the most part, overalls, boots and gloves.

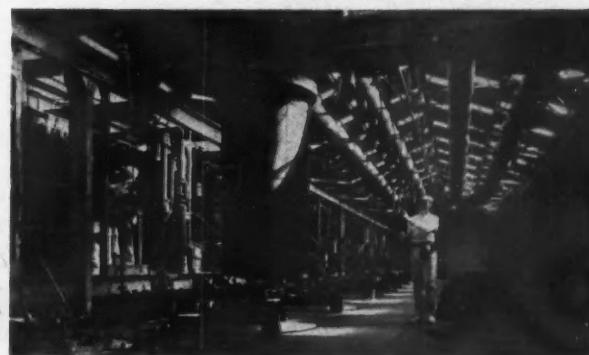
First drums of uranium ore were sampled in January 1948 and in the following October the first uranium rods were produced from material which had passed completely through the works. In August 1954 the first uranium concentrate was processed. The tributyl phosphate purification process was begun in July 1956 and by April 1957 the changeover to magnesium reduction was completed. The new processing plant was opened last June by Lady Plowden.

The Original Ore to Billet Process. Pitchblende, used as the starting material was crushed and reduced to $\frac{1}{2}$ in. mesh. The crush material was then converted into a slurry by mixing with water in a ball mill. This slurry was fed to a heated stainless steel tank together with nitric and sulphuric acids, which dissolved the uranium and left the bulk of the impurities, including the radium, in suspension. The material was then pumped through filter presses to remove the solids, and the crude uranyl nitrate solution was obtained from which uranium was precipitated by the addition of hydrogen peroxide. The uranium per-

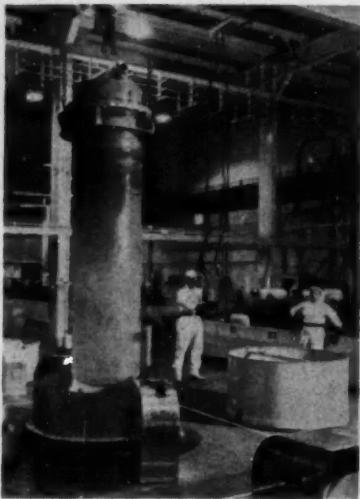
oxide thus formed, was collected on filter presses.

The filter cake was dissolved in warm nitric acid and the solution evaporated until uranyl hexahydrate formed. This was fed into an extractor where an ether stream extracted most of the uranium, leaving the impurities in the aqueous solution, which sank by gravity, to the bottom of the vessel, from which it could be run off. The uranyl nitrate was washed back out of the ether into water and treated with ammonia, when uranium was precipitated as ammonium diuranate.

Nutsch Filters. The liquor containing the suspended ammonium diuranate was run off through Nutsch filters. The ammonium diuranate paste was loaded into circular trays which were of two types, one with a hole in the centre and the other with a series of holes round the rim. The trays were stacked alternately in a container which was lowered into an electric furnace sunk into the floor and connections were made to gas lines and an effluent pipe. The furnace was first heated for some hours to decompose the ammonium diuranate, leaving uranium trioxide in the trays. Hydrogen (produced on the plant by electrolysis of

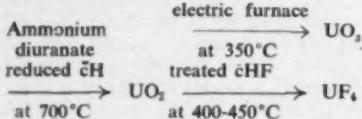


Dryway reactor being loaded into a furnace at Springfield



In the Mg reduction of uranium tetrafluoride to uranium metal, one of the red-hot reactors is shown being removed from the furnace to the cooling bay

caustic soda) was passed through, reducing the trioxide to dioxide, and then anhydrous hydrofluoric acid which converted the dioxide to uranium tetrafluoride.



Arrangement of the holes in the trays ensured that the gases effectively covered the whole of the material in each tray, flowing from the centre to the edges on one tray and in the reverse direction on the next. As the three compounds produced during the process are markedly different in colour (i.e.—the trioxide is orange-coloured, the dioxide a brownish colour and the tetrafluoride a turquoise green), it was a simple matter to determine if the final conversion had been completed.

At the time that the process was origin-

ally worked out, there were two alternatives for this stage. Since the other was carried out with solutions, the two methods became known as the Dryway and Wetway methods. This section of the plant is the Dryway section.

After removal from the furnace and cooling, the containers were dismantled and the trays emptied. The uranium tetrafluoride was mixed with chippings of calcium metal and loaded into a cast iron crucible lined with calcium fluoride. This was placed in a firing chamber, sealed in and the mixture ignited remotely. In an exothermic reaction the calcium combined with the fluoride atoms and left uranium in metal form. The molten uranium sank to a cavity in the bottom of the crucible lining, where it formed a cylindrical billet, with a thick layer of calcium fluoride slag on top.

Uranium Processing in the New Plant. The new process uses concentrate (crude uranium oxide, U_3O_8 as calcine magnesium diuranate or calcine alumina diuranate) obtained at the present time from three sources of supply—Canada, South Africa and recently from the Mary Kathleen Mine, Australia. This eliminates the peroxide precipitation stage of the original process. If at any time the starting material changes, it will be given a separate pretreatment to convert it to the approximate composition of the concentrate. Samples are taken of the concentrate to determine uranium content (not less than 60% U_3O_8).

Drums of concentrate are taken up to the tipping area as required from the store below. The lids are removed and the drums pass into the totally enclosed tipping cubicle where the concentrate is tipped into a hopper with a screw feed to the dissolvers. These are large stainless steel tanks (three in number of about 1,000 gall. capacity) fitted with steam heating coils from four stirrers. The concentrate is dissolved in nitric acid. Underneath the floor carrying the primary dissolvers and filters the various necessary intermediate stages are performed. An insoluble sludge, mainly composed of silica, has to be removed. This is sampled to determine whether it contains appreciable quantities of uranium. If uranium is found, this is recovered in the recovery line.

After the four stages of dissolving, the solution is cooled and fed to four vacuum rotary filters consisting of large rotating horizontal drums of fine mesh covered with a precoat of diatomaceous earth. Solid impurities extracted by these filters are resuspended in tanks on the floor below. The slurry is then passed through a secondary rotary filter to ensure that all traces of uranium (as uranyl nitrate) are removed.

Purification of the uranyl nitrate solution is effected by counter-current solvent extraction with a solution of tributyl phosphate (TBP) in colourless kerosene in mixer-settler units arranged in a cascade. Because of its greater solubility in TBP, the uranyl nitrate collects in the solvent phase, leaving the impurities in the aqueous phase. Springfield claim that removal of uranyl nitrate from the aqueous phase can be made almost complete. Tributyl phosphate emerges containing uranyl nitrate in solution, passes through a heating unit and the uranyl nitrate is then washed out of the solvent by a weak nitric acid solution.

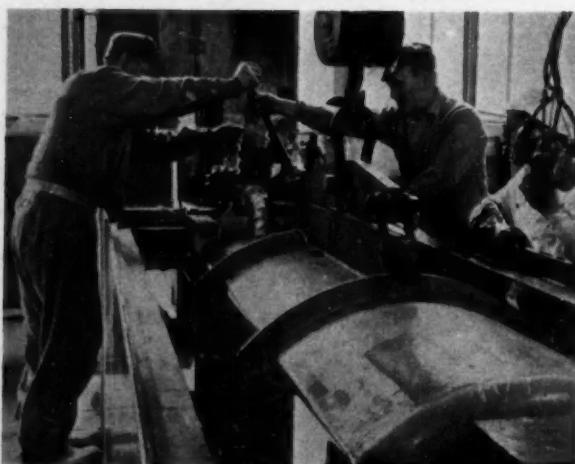
The pure uranyl nitrate solution from the solvent extraction process is held temporarily in stock tanks prior to its being pumped to the next part of the line where the uranyl nitrate is converted to uranium tetrafluoride. The solution is kept circulating in these tanks by pumps to prevent settling.

Reverts to Old Process

It is at the above stage that the process reverts to the old process. The uranyl nitrate solution is treated with 0.88 ammonia to precipitate the uranium as ammonium diuranate. After the ammonium diuranate settles the mother liquor is siphoned off and the product is washed four times with pure water—the water being siphoned off after every washing. The ammonium diuranate is filtered from the remaining water in Nutsch filters, using twill filter cloths, leaving the ammonium diuranate as a 'cake'. This compound is then put through the Dryway plant to emerge as uranium fluoride. Ultimately it is planned to carry out this part of the process by fluidised bed methods. Development work on this is reported to be at an advanced stage and the section of the new plant in which this will be done is at present under construction. When this section is in operation, the uranyl nitrate solution from counter-current extraction will pass to a precipitator unit and then through the trioxide and dioxide to the uranium tetrafluoride.

Reduction of UF_4 . In the old process calcium metal was used with the uranium tetrafluoride in the reduction to uranium metal. It was realised, however, that magnesium was a cheaper alternative reducing agent to calcium but considerable development work was necessary before a satisfactory process was produced. In April 1957, the changeover to magnesium reduction was finalised. The mixture of uranium tetrafluoride and magnesium turnings is pelleted before being packed into the furnace where reduction of the tetrafluoride to uranium metal takes place.

Uranium tetrafluoride and magnesium turnings are delivered to an automatic



A 'Hex' bottle being connected to the pipeline from the fluorination reactor. The uranium hexafluoride leaves the reactor in gaseous form and condenses to the solid form inside the water-cooled vessel

weighing and mixing machine which feeds the pelleting press. This is also automatic and ejects the pellets (of 3 kilogrammes weight) to a conveyor which delivers them to the furnace reactor breakdown and loading section.

The reactor consists of a long steel cylinder containing a sectioned graphite lining. The pellets of uranium tetrafluoride-magnesium are built up inside the graphite lining and the whole assembled in the reactor. The reactor is passed into the adjacent furnace bay where it is placed in an electric furnace in which the reduction reaction takes place. After cooling the reactor is returned to the furnace bay, dismantled and the slag (magnesium fluoride) and uranium billet removed. The uranium is better than 99% pure. Slag adhering to the billet is removed by placing the billet in hot ammonium chloride solution, then nitric acid and finally removing all traces of slag in a vacuum blast machine.

From Billet to Fuel Element. First step in the conversion of the uranium metal into a fuel element is to melt the billet plus pieces of previous casting headers, rejected rods, etc. (weight about 500 to 550 Kg.), in a vacuum furnace heated by a high frequency induction current (vacuum $\frac{1}{2}$ cm. Hg) for about 1½ to 2½ hours, and pour the molten metal into cylindrical moulds. A complete melt is poured at once by removing a spigot in the base of the furnace. After cooling the mould assembly is dismantled and the rods are taken into the bond store, each cast being kept separate.

Machining Operations

The rods then undergo a succession of machining operations to bring them to the desired design, with heat treatments included to produce a satisfactory metallurgical condition to minimise distortion during irradiation. In particular, the uranium rods are now subjected to beta-quenching which is designed to reduce the grain size of the uranium metal and prevent surface wrinkling. Beta-quenching is carried out by passing the rod through a variable speed motor at a frequency of 500 and at about 800°C.

After this treatment the rods pass to the alpha-annealing apparatus—again part of the heat treatment designed to reduce grain size of the uranium metal. An assembly of 30 rods at a time is placed in a container prior to undergoing (a) evacuation of the container and filling with argon (produced at the plant); (b) removal to furnaces to be heated and held at constant temperature (550°C); (c) returned to another bay for controlled air cooling (about 1 to 2 hours).

Machining operations are entirely conventional and are carried out on standard lathes or centreless turners, the only precautions taken being the provision of an exhaust system away from the operator and a copious supply of coolant to prevent ignition of the swarf and minimise the risk of particles escaping into the atmosphere. Turnings are collected, cleaned and added to subsequent casting charges. A specially designed machine cuts the rod to slightly more than the required length (40 in.), cuts a hanging

groove to facilitate later operations and stamps the number of the rod on to the end.

Finally the rods, inspected and checked for dimensional accuracy are sent for assembly into fuel elements. Early fuel elements, for BEPO and the Windscale reactors, used aluminium as the canning material, but with the higher operating temperatures in the Calder Hall reactors, aluminium was unsuitable. However, the change from air to relatively inert carbon dioxide as the cooling gas in these reactors has eliminated the fire risk associated with magnesium and the cans for Calder Hall, Chapel Cross and the reactors of the civil nuclear power stations now under construction are of a magnesium alloy known as Magnox (a mixture of magnesium, aluminium and some beryllium).

The Magnox cans arrive at Springfield and consist of open-ended finned tubes. After degreasing in trichloroethylene they are examined and comprehensively gauged. A cap is inserted in one end and argon-arc welded in position automatically. After the weld is tested, the uranium rod is loaded into the can. To prevent uranium contamination of the outside of the fuel elements no uranium is handled or allowed uncovered in the fuel element assembly part of the line. The second end of the can is then sealed

by inserting a cup and welding in the same way as the first end. The fuel element can is also evacuated and filled with helium to aid subsequent leak detection.

The resulting cartridge is pressurised using oil at 200 to 250°C at 10,000 p.s.i. to ensure that perfect thermal contact between the can and the uranium rod is obtained. This is checked by X-raying the cartridge. To determine whether there is any leak in the canned fuel element, the rods are examined by a mass spectrometer for leakage of helium.

In the final assembly section the Magnox can receives its final clean using a Mullard ultrasonic washing machine with 1½% citric acid solution, followed by a water wash and drying. The fins of the can are then straightened and each can is packed in a polythene sleeve and placed three at a time, in special despatch boxes.

At Springfield uranium hexafluoride for the gaseous diffusion plant at Capenhurst is also processed. After the fuel elements have been irradiated they are processed at Windscale to separate the fission products and plutonium. The chemically unchanged uranium in which the proportion of the fissile 235 isotope is reduced, is returned to Springfield as a solution of uranyl nitrate, where it is converted to uranium hexafluoride for re-enrichment at Capenhurst.

Sir John Cockcroft to Head New Cambridge Science College

FIRST Master of Churchill College will be Sir John Cockcroft, member for research of the U.K. Atomic Energy Authority and former director of the Atomic Energy Research Establishment, Harwell. Future Masters are to be appointed by the Crown on the advice of the Prime Minister; Sir John was invited by his fellow trustees of the college to be its first Master.

A site of about 41 acres is being purchased on Madingley Road, Cambridge, from St. John's College. It is hoped that one residential block and other main buildings will be ready by October 1961. So far the trustees have received, or have been promised, £2,832,000 and with that sum they feel they can open the college on a workable, but limited basis. To be sure that their expenses would be covered a further £670,000 would be needed; to make the college even more effective the trustees need a further £1 million on top of that.

The third list of contributions includes sums from the following:

UNDER DEED OF COVENANT

Per Year for 10 Years: £1,000, Albright and Wilson Ltd. and associated companies; Fisons Ltd. £100, Associated Chemical Co. Ltd.; Goigy (Holdings) Ltd. £99 and under; Forestal Land, Timber and Railways Co. Ltd.; Manchester Oil Refinery Ltd.

Per Year for 7 Years: £1,500, Reckitt and Colman Holdings Ltd. £1,000, Laporte Group of Industries, Fullers' Earth Union Ltd.; Regent Oil Co. Ltd. £750, C. C. Wakefield and Co. Ltd. £548, Foster Wheeler Ltd. £500, Cape Asbestos Co. Ltd. £348, Hickson and Welch Ltd. £286, Clayton Aniline Co. Ltd. (of C.I.B.A. Group). £250, Calico Printers' Association Ltd.; Goodlass Wall and Lead Industries Ltd. £200, Baker Perkins Ltd.; A. Boak Roberts and Co. Ltd.; Steetley Co. Ltd. £182.

British Industrial Plastics Ltd. £174, Midland Tar Distillers Ltd. £143, Ciba (A.R.L.) Ltd.; Ciba-Clayton Ltd.; Ciba Laboratories Ltd. £107, Whessone Ltd. £100, Cambridge Instruments Co. Ltd. £99 and under, B.B. Chemical Co. Ltd.; W. J. Bush and Co. Ltd.; Cooper, McDougall and Robertson Ltd.; Hardman and Holden Ltd.

SINGLE PAYMENTS

£10,000, Thomas Hedder and Co. Ltd. £1,000, Smith and Nephew Associated Companies Ltd. £105, Anchor Chemical Co. Ltd.; Hilger and Watts Ltd.; Woodall-Duckham Construction Co. Ltd.

New Economic Insecticide Marketed in U.K.

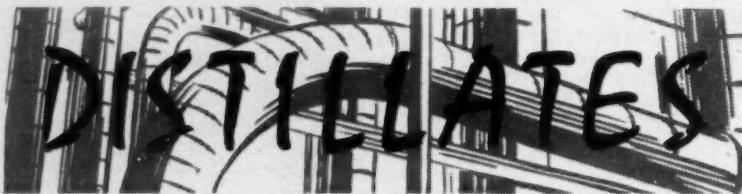
THE claim that it is the most active aphicide yet discovered, as well as one of the most economical, is made for a new British systemic insecticide.

In trials the insecticide, which is marketed jointly by Leek Chemicals Ltd., Bridge End Works, Leek, Staffordshire, and Associated Fumigators Ltd., has proved lethal to all kinds of aphids, including the woolly aphid, the peach potato aphid and blackfly.

It is being used initially on sugar beet and is stated to be unlikely to lead to a hazard to consumers of either the sugar, molasses, beet tops or pulp. It is hoped to get clearance on beans, cabbage and other vegetables.

Unlike most other systemics, it is not an anticholinesterase inhibitor. It has contact and fumigant properties.

It has proved lethal at a concentration of one part in 20,000 parts of water and at a low dosage will cost about 6s. an acre to apply.



★ WHEN I visited the new Springfield Works of the U.K. Atomic Energy Authority last week, I was interested to hear Sir William Cook indicate that production of fuel elements was likely to become one of Britain's largest industries in the next few years. There are, of course, considerable possibilities for exporting uranium fuel elements. There will be plenty of competition although at present the only competitor in the field is the U.S.

This month, Mallinckrodt Chemical Works, St. Louis, set the ball rolling in advancing U.S. commercial development of atomic energy by creating a new wholly-owned subsidiary, Mallinckrodt Nuclear Corporation. The new Company is to take over the parent company's \$2 million plant at Hematite, Mo. It has already processed more than 150,000 lb. of enriched uranium hexafluoride into nuclear reactor fuel elements in the past two years. This material has been sold to more than 80 reactor designers, builders and owners, including all reactor projects completed or under construction in the U.S.

Also in the running are Allied Chemicals' General Chemicals Division, which have just begun to operate their uranium hexafluoride plant at Metropolis, Ill.

★ OXYGEN production is booming in the U.S. and is expected to expand by 33% this year. This increase over the record 1958 figure does not include oxygen used in the Government's missile programme. Missiles may use as much as 20,000 million cu. ft. of oxygen bringing total national consumption to more than 100,000 million cu. ft.

If production is expanding so also is competition; the 100 U.S. oxygen producers already operate more than 375 plants with a total capacity of 130 to 140,000 million cu. ft. And 26 more plants are scheduled for construction before the end of 1959! Main reason for the growth in new plants is to supply the steel industry; the average ingot-ton of steel produced in the U.S. required 250 cu. ft. of oxygen gas; this requirement may well climb to six times this amount by the early 1970's.

And to think that at the A.E.A. uranium processing factory last week I was told that the oxygen produced is allowed to go to waste in the atmosphere!

★ I.C.I. with one of the best accident-free records of any industrial concern, have as a target a frequency rate of 0.25 (based on the number of accidents causing loss of time which have occurred for every 100,000 hours work—the normal working life of the individual).

In 1946 the rate was slightly under 3.0. Since then it has steadily declined until this year, to date, it is 0.441.

Now the company has produced a dramatic colour film entitled 'The Human Factor', a safety film with a difference. Although it dramatises an 'accident' at the Castner-Keller works, there is no 'villain of the piece' on whom all the blame is heaped. The film was produced by the company's own film unit in an attempt to analyse the causes of an industrial accident and to show that it was caused by a number of different circumstances all happening at once.

The film, which should prove of great value when shown round the I.C.I. divisions, is also being made available to other industrial organisations.

★ AFTER long trials, Pfizer Ltd., Folkestone, Kent, have introduced their influenza virus polyvalent vaccine. This new vaccine contains three different strains of inactivated type A virus and one strain of inactivated B virus. The Asian strain of type A virus is present in a concentration of twice that of the other components.

I saw the production unit at Pfizer's Sandwich plant last spring but at that time visitors to Sandwich were not allowed near the unit nor told anything about it. The reason was understandable—the company did not want to announce their interest in the field prematurely. They felt that there had already been enough premature advance publicity and were determined to wait until their vaccine had been 100% cleared by their own test-house as well as by the stringent Ministry tests. Even questions in Parliament last year would not bring any statement from the company.

★ FEARS that there might be a toxic hazard associated with the use of continuous-flow, heat-volatilised insecticidal aerosols have been expressed from time to time since these aerosols were introduced into the U.K. in 1946. These fears have resulted from U.S. experiences.

I have been reassured that the aerosols in use in the U.K. are, however, unlikely to give rise to such risks. Writing in *The Lancet* (1958, II, 1177) Drs. A. Howard Baker and F. H. Whitney, of Avebury Research Laboratories, Goring-on-Thames, and Dr. A. N. Worden, Nutritional Research Unit, Huntingdon, state that over a period of 12 months they have kept a record of the output of dicophane (DDT) lindane mixtures, aldrin or azoxybenzene from units installed in five animal laboratories.

Their results show that there is a range of doses of dicophane/lindane in which

it appears possible to combine effective fly control with safe operation. Upper limit of the range is estimated between 0.53 and 0.75 g.d.t. (gm. per day per 1,000 cu. ft.) for budgerigars and perhaps for mice. Upper limit for dogs lies above 0.22 g.d.t.; for rats, above 0.53 g.d.t. and for humans, above 0.75 g.d.t. when exposed intermittently for a total of at least 206 hours.

Vaporised aldrin at 0.50 g.d.t. and azoxybenzene at 0.43 g.d.t. in the control of horticultural pests was not seen to offer any toxic hazards to man, budgerigars, mice, hamsters or guinea pigs.

★ THE NAME of Carnegie is synonymous with philanthropy; the story of the Scottish boy who became an American multi-millionaire is known throughout the world. Last week I learned of an English Carnegie family which sold its pharmaceutical business to an American company and which will devote the proceeds of the sale to the furtherance of Christian work.

Mr. Donald Carnegie, who became chairman of Carnegies of Welwyn on the death of his father in 1954, has sold the company to the Rexall Drug Co., Los Angeles. Two of his sons who were connected with the company will, it is reported, now study for theological degrees. A third son is at the Royal School of Church Music.

The family which came originally from Scotland, has a background of staunch religious conscience. In 1955, the Welwyn Fellowship House, an inter-denominational meeting place was built in memory of the founder, Robert Carnegie.

The company became the largest producers of quinine in Britain; it also makes bismuth salts, strychnine and adrenaline.

★ UNDERGROUND gasification tests carried out by the National Coal Board at Newman Spinney, near Chesterfield, are to end because the commercial prospects "do not justify further expenditure". As considerable development remains to be done, N.C.B. has therefore decided to terminate their development contract with Humphreys and Glasgow.

Some further trials will take place, however, before production of gas and electricity at the site stops in the early summer. Overall cost of the experiments is stated to be about £1 million.

Within the next few weeks, the N.C.B. hopes that full production of the pilot station will be achieved from gas provided by two different systems of experimental gasification.

Humphreys and Glasgow report that they might be able to introduce the process in some of those countries known to have coal deposits suitable for the process. Under favourable geological conditions, the company states "gas produced underground could be competitive with any other form of power station fuel".

Alembic

D.C.L.'S CHLOROPRENE PATENTS

Series Deals with Preparation of and Intermediates for Chloroprene

DURING the last seven months, the Distillers Co. Ltd., have been granted a series of patents, which suggest that the company has more than a passing interest in the commercial production of chloroprene. As yet, however, there has been no indication from the company that it has any plans in this direction.

The preparation of chloroprene and in particular its production by the dehydrochlorination of 3:4-dichlorobutene-1 was detailed in the British Patent No. 798,205 (16 July 1958). This method of preparation whereby 3:4-dichlorobutene-1 is heated with an aqueous solution of an alkali is well known. Hitherto, however, theoretical yields of chloroprene have not been attainable owing to the ease with which the chloroprene polymerises during the reaction giving products which for the most part cannot be distilled. Patent 798,205 gives details of an improved process in which the formation of chloroprene polymers is appreciably reduced. The heating of 3:4-dichlorobutene-1 with an aqueous solution of alkali is carried out in the presence of a salt of picric acid. The presence of picric acid in the form of a salt such as calcium or sodium picrate effectively inhibits polymerisation of chloroprene and as a result greatly improved yields are obtained. The concentration of picrate used is not critical; concentrations of 0.1 to 1.0% by weight based on the volume of the reaction medium, and calculated as picric acid, are suitable.

For preference the reaction is carried out by introducing 3:4-dichlorobutene-1 into a solution of alkali, such as a 10 to 20% aqueous sodium hydroxide solution, containing the picrate salt, with stirring or agitation. The solution is maintained above the boiling point of chloroprene, i.e., about 60°C., which is distilled off as it is formed.

Process Examples

Examples are given to illustrate the process. The apparatus for the dehydrochlorination of 3:4-dichlorobutene-1 consisted of a reaction vessel fitted with a stirrer and a distillation column. Two hundred and thirty-eight parts by weight of sodium hydroxide and 2 parts by weight of picric acid dissolved in 950 parts by volume of water were charged to the reaction vessel. This gave a 20% wt./wt. solution of caustic soda in 26% excess over the stoichiometric requirement for the 3:4-dichlorobutene-1. The reaction mixture was heated to 90°C and 592 parts by weight of 3:4-dichlorobutene-1 were fed in over a period of between 1 and 2 hours. Two fractions were distilled off. A yield of chloroprene of 94.3% based on 3:4-dichloro-

butene-1 was obtained. The percentage conversion of 3:4-dichlorobutene-1 to polymeric products was nil.

In British Patent No. 798,347 (published 16 July 1958) Distillers dealt with the stabilisation of chloroprene. Under the influence of light or heat chloroprene has a tendency to polymerise. Also,

The series of patents taken out by the Distillers Co. Ltd., and reviewed here deal with the following aspects of the production of chloroprene:

- **Production by dehydrochlorination of 3:4-dichlorobutene-1 (798,205).**
- **Stabilisation of chloroprene (798,347).**
- **Dehydrochlorination of 1:2:3-trichlorobutane (805,816).**
- **Preparation of dichlorobutenes (798,393 and 800,787).**

Polymerisation is particularly liable to occur in purification processes as when impure chloroprene is being distilled. Even on standing, chloroprene is liable to polymerise. Distillers' F. T. Bellringer, in the above mentioned patent, reports that the adventitious polymerisation of chloroprene can be effectively inhibited with nitric oxide, both in the liquid phase and in the vapour phase.

Especially noteworthy is the fact that nitric acid inhibits polymerisation in the vapour phase, since many of the conventional stabilising agents, although effective in preventing the polymerisation of the liquid chloroprene in the kettle of a still, do not prevent the formation of polymer from chloroprene in the vapour phase in the still itself. "By mixing the chloroprene vapour in the still with traces of nitric oxide, preferably in the presence of an inert gaseous diluent such as nitrogen, carbon dioxide, helium, or argon, such polymerisation is substantially prevented.

"Suitably, nitrogen containing traces of nitric oxide is bubbled through the kettle or is introduced into the distillation vessel directly above the surface of the distillant. In addition to preventing polymerisation in the vapour phase small amounts of nitric oxide dissolve in and stabilise the distillate".

The amount of nitric oxide which is effective is not critical, it is reported. In the case of liquid chloroprene, a small amount of nitric oxide is sufficient to prevent polymerisation. In the case of gaseous chloroprene, again only traces of nitric oxide are required. "In general amounts of about 0.01% by volume of nitric oxide are sufficient to stabilise the chloroprene".

It has been found that chloroprene stabilised with nitric oxide can be polymerised under the normal polymerisation reaction conditions without having first to remove the nitric oxide stabiliser.

In an example quoted in the patent, impure chloroprene obtained by the alkaline dehydrochlorination of 3:4-dichlorobutene-1, containing as impurities traces of 1-chlorobutadiene and 3:4-dichlorobutene-1 was dried and fractionated at normal pressure in the absence of light through a 5 ft. by 1 in. Fenske helices packed column; 0.25% of a 50/50 mixture of *p*-tert. butyl catechol and phenothiazine was added to the kettle. Nitrogen containing nitric oxide was passed into the kettle and the chloroprene was fractionated at a 10/1 reflux ratio over a period of 20 hours. At the end of this time, when the kettle residue was very small, polymer formation was not detectable.

When the same impure chloroprene was fractionated in a 3 ft. by 1 in. Fenske helices packed column with 0.25% of said mixture of *p*-tert. butyl catechol and phenothiazine in the absence of nitric acid polymer was observed in the kettle after 2 hours and after 6 hours the loss due to polymer formation was 25% of the charge.

Dehydrochlorination

Under the title, 'Dehydrochlorination process' a patent (British Patent No. 805,816) published 10 December last year, has been granted to E. G. E. Hawkins of Distillers. This patent relates to the production of chloroprene (2-chlorobutadiene) and hydrogen chloride using as starting materials chlorine and a straight chain hydrocarbon containing four carbon atoms such as butene-1, butene-2 or butane. In particular, it relates to the production of chloroprene precursors and hydrogen chloride by the dehydrochlorination of 1:2:3-trichlorobutane. (Mixtures of trichlorobutanes including 1:2:3-trichlorobutane are obtainable from butene-1, butene-2 or butane by chlorination).

Various processes have been proposed for the production of chloroprene and hydrogen chloride from various chlorinated derivatives of butane or the butenes, notably by pyrolysis of 2:2:3-trichlorobutane and of certain dichlorobutenes. However, initial chlorination of the straight chain hydrocarbon gives rise to chlorinated products which cannot, by pyrolysis, be converted, or are only converted in small yield, to chloroprene and hydrogen chloride. Chlorination of butene-2 to produce trichlorobutanes, for instance, gives rise to 1:2:3-trichlorobutanes as well as the desired 2:2:3-trichlorobutanes and chlorination of butene-1 to a trichlorobutane gives as chief product the 1:2:3-isomer. Direct chlorination of butane gives rise to mixed trichlorobutanes.

The present process described in this patent provides for the conversion of 1:

2:3-trichlorobutane into chloroprene precursors. By this is meant a chlorine derivative of the butenes which, on pyrolysis or isomerisation followed by pyrolysis, gives primarily chloroprene and very little unwanted 1-chlorobutadiene.

Such chlorine derivatives of the butenes are 1:2-dichlorobutene-2, 2:3-dichlorobutene-1, and 1:3-dichlorobutene-2. The patent also provides a method for converting 1:2:3-trichlorobutane into chloroprene and hydrogen chloride with the minimum formation of unwanted by-products. Finally a process is given, the use of which can improve overall yield of chloroprene from trichlorobutanes obtained by the chlorination of butene-1, butene-2 or butane.

In this latest Distillers' process, 1:2:3-trichlorobutane is converted into hydrogen chloride and chloroprene precursors, by heating in the presence of a catalyst of the hydrochloride or a quaternary ammonium chloride salt derived from an amine having a pK value in the range 3.0 to 9.0 and a boiling point at atmospheric pressure between 110° and 250°C.

C—N Bond

In this specification 'amine' refers to derivatives of ammonia in which one or more of the hydrogen atoms is replaced by an organic radical thus forming a C—N bond. Thus compounds in which the N atom forms part of a cyclic system, e.g. pyridine and piperidine, are included. The term 'pK' of an amine is defined as $\log_{10} 1/K$, where K is the basic dissociation constant of the amine expressed as the product of the concentrations of the ammonium and hydroxyl ions divided by the total concentration of unionised material present as either the free base or hydrate.

It is reported that either pure 1:2:3-trichlorobutane can be used or the reaction can be carried out using as starting material a mixture of isomeric trichlorobutanes. Thus, butane, butene-1 or butene-2 may be chlorinated to give 2:2:3-trichlorobutane and 1:2:3-trichlorobutane. 2:2:3-Trichlorobutane is then dehydrochlorinated to give chloroprene and the 1:2:3-trichlorobutane is treated as mentioned above to give chloroprene precursors. These latter are then dehydrochlorinated to give chloroprene.

Tertiary amines, the patent states, give rise to particularly good catalysts. Preferred catalysts are derived from α - and γ -picolines, 2:4:6-collidine, 5-ethyl-2-methyl-pyridine and tri-n-butyl'am's. Other suitable amines include pyridine, quinoline, *N*-ethylpiperidine and cyclohexylamine. The amine hydrochlorides are the preferred catalysts. These or the quaternary ammonium chloride salts act as true catalysts in this present process and each molecular proportion of catalyst effects the dehydrochlorination of many molecular proportions of 1:2:3-trichlorobutane. The amine effects the dehydrochlorination reaction but no free hydrogen chloride is given off until a sufficient quantity of hydrogen chloride has been produced to convert the amine into its hydrochloride. It has been found that while many amines can effect the stoichiometric dehydrochlorination of 1:2:3-trichlorobutane, unless they have the

characteristics of amines described above, their formed hydrochlorides do not catalyse the production of free hydrogen chloride and chloroprene precursors.

The catalytic dehydrochlorination reaction is effected by heating the reaction mixture, preferably to a temperature above 150°C. It is convenient to carry out the reaction by heating the reaction mixture under reflux and consequently, under atmospheric pressure, temperatures in the range 150° to 220°C or above being used. The rate of reaction is increased by raising the temperature. As the temperature of the mixture is increased it may become necessary to raise the pressure in the reaction system "so that part at least of the reacting material is maintained in the liquid phase." An inert solvent for 1:2:3-trichlorobutane and/or catalyst can be employed. This solvent should have a sufficiently high boiling point to prevent it being distilled out of the reaction mixture during the process. Such solvents are diphenyl ether and chloronaphthalene.

Rate of dehydrochlorination, it is noted, is partly dependent on the amount of catalyst used. It is often advantageous to add a high proportion of catalyst, for instance, 25% or 50% by weight or more, to the initial reaction mixture in order to obtain a suitably fast rate of reaction.

Batch or Continuous

This latest process can be carried out batchwise or continuously. A suitable apparatus for continuous working consists of a fractionating column above a heated reaction vessel which can be charged with a mixture of 1:2:3-trichlorobutane and the amine catalyst. The mixture is boiled so that it refluxes in the column and gaseous hydrogen chloride is separated from the liquid reflux in the still-head at the top of the column and led away. When the head temperature fails to near the boiling point of the produced dichlorobutenes (about 125°C), a part of the reflux is bled off as product at such a rate that the head temperature does not rise above a value predetermined by the degree of conversion required. Control of the rate of take-off may be manual or by means of an automatic device operated by a temperature-sensitive element in the stillhead. At the same time, 1:2:3-trichlorobutane is pumped into the reaction vessel at such a rate that the volume of its contents remains approximately constant.

An alternative and preferred arrangement, it is stated, is to lead the vapours from the reaction vessel into the column at a point several plates above the base. A reboiler at the base of the column is then necessary to maintain reflux in the lower part. The excess liquid in the reboiler (mainly unchanged trichlorobutane) is returned to the reaction vessel by means of a pumping or overflow device.

A certain amount of polymer may form in the reaction vessel after a long period of running. To eliminate this and any other undesirable substances which may accumulate in the reaction mixture, it is recommended that intermittently or continuously a proportion of the liquid

should be drawn off, recovering therefrom trichlorobutane, dichlorobutenes and the catalyst and returning these to the reaction vessel while rejecting the residual material. Any amine lost during the above process is replaced so as to maintain the desired concentration of catalyst in the reaction mixture.

Dichlorobutenes are produced, but surprisingly substantially no dichlorobutene which on pyrolysis, give 1-chlorobutadiene. Chief products of the dehydrochlorination of 1:2:3-trichlorobutane using the process outlined above are 1:2-dichlorobutene-2 and 2:3-dichlorobutene-1, with, in some cases, small amounts of 1:3-dichlorobutene-2. As these have lower boiling points than trichlorobutane, they can be distilled off from the reaction mixture as they are formed, or taken from the top of a reaction zone fitted with a reflux condenser or column. The gaseous hydrogen chloride formed in the reaction may be recovered by any suitable means. The hydrogen chloride is produced in substantially anhydrous form.

Particularly Useful

Hawkins reports that the reaction described is particularly useful when applied to the production of chloroprene from butene-1 or butene-2. These butenes can be converted by chlorination into trichlorobutanes as a first step in the production of chloroprene.

Practically, it is stated to be advantageous, particularly when the chlorination is to be carried out continuously, to chlorinate to an average chlorine content less than that required for complete conversion and then to distil the product continuously and return the lower boiling dichlorobutanes to the chlorination reactor. Thus mixtures of trichlorobutenes substantially free from over-chlorinated products are obtained. The formation of 2:2:3:3-tetrachlorobutane should be prevented as this compound is difficult to separate from 1:2:3-trichlorobutane by distillation. It is stable, however, under the conditions of the present invention. It is solid up to 180°C and can, therefore, cause blockages inside the apparatus, particularly if it is allowed to accumulate when operating the process continuously. It can be separated from 1:2:3-trichlorobutane by treatment with thiourea according to co-pending British Application 31855/55 (Serial No. 805,817).

The mixture of 2:2:3-trichlorobutane and 1:2:3-trichlorobutane obtained by chlorination of butane-2 is readily separated into its components by distillation and two components treated separately as outlined above. It has been found somewhat wasteful to subject 1:2:3-trichlorobutane to the present process when it is present in a crude chlorination reaction mixture containing a substantial amount of 2:2:3-trichlorobutane.

On subsequent pyrolysis the mixture of 1:2-dichlorobutene-2, 2:3-dichlorobutene-1 and small amounts of 1:3-dichlorobutene-2 are converted in known manner to hydrogen chloride and chloroprene substantially free from 1-chlorobutadiene. 2:3-Dichlorobutene-1 gives a greater yield of chloroprene on pyro-

lysis than do the other dichlorobutenes formed by the process noted above. The ultimate yield of chloroprene may therefore be increased by isomerising the 1:2-dichlorobutene-2 to 2:3-dichlorobutene-1 under the influence of heat alone, i.e., pyrolysis conditions at 450° to 650°C (preferably 500° to 530°C), or under the influence of copper or a copper chloride catalyst under reflux conditions.

Twelve examples are given illustrating the conversion of 1:2:3-trichlorobutane into dichlorobutenes using the above mentioned process.

Two patents have been granted to Distillers relating to dichlorobutenes. On 23 July last year, British Patent 798,393 entitled 'Preparation of dichlorobutene' was published. The other, British Patent 800,787, published 3 September 1958 is entitled 'Preparation of 3:4-dichlorobutene-1'.

Dichlorobutenes

The first patent covers the production of dichlorobutenes by active chlorination of butadiene in the vapour phase at an elevated temperature "in the presence of an alkane and/or an alkene having four carbon atoms." The alkanes and alkenes present during additive chlorination of butadiene in this process are butane, isobutane, butene-1, butene-2 and isobutene. For preference butene-1 and/or butene-2 or mixtures of butene and butane are used, since these are readily available as fractions obtained in the distillation of cracked petroleum. In the present reaction the alkane or alkene is substitutively chlorinated; butene is converted into mono-chlorobutene and butane is converted into mono-chlorobutane.

It is claimed that chlorination of mixtures of butadiene with these hydrocarbons results in higher yields of dichlorobutenes than can be obtained when butadiene is chlorinated alone. It has also been found that in the mixed chlorination the formation from the butadiene of undesirable by-products, particularly 1-chlorobutadiene and high-boiling compounds, is appreciably reduced. Also, a substantially higher reaction temperature, and therefore a much higher reaction rate of butadiene chlorination giving economy in reactor size, may be used than is possible when butadiene is chlorinated alone.

A further advantage of using butene is that the small amount of 3:4-dichlorobutene-1 normally found in the substitutive chlorination of butene is recovered with the dichlorobutenes formed from the butadiene.

The proportion of alkane and/or alkene to butadiene used in the chlorination is not critical and any ratio may be used. However, the effect of alkane or alkene in inhibiting side reactions is stated to be less pronounced when it is present only as a small proportion of the total hydrocarbon in the reaction mixture.

A suitable source of the starting material for the process is crude C₄ stream. This stream is subjected to extractive distillation with aqueous furfural, and n-butane, isobutane, butene-1 and isobutene are taken off overhead and a mixture of butene-2 and butadiene are removed as residue. Mixtures of butene

and butadiene containing n-butane and small amounts of iso-compounds (e.g., isobutane and isobutene) may also be available. "Such mixtures are likely to be considerably cheaper than their components in the pure state."

It is preferred "to use molar ratios of total hydrocarbon: chlorine with excess of hydrocarbon" in order to suppress production of derivatives containing more than the desired amount of chlorine. It is also desirable "to preheat the hydrocarbons to near reaction temperature before mixing them with chlorine. Rapid and efficient mixing of the reactants is necessary if optimum yields are to be achieved."

The reaction is carried out with advantage in the presence of an inert gas or diluent (between 5% to 50% by volume of the mixture) such as nitrogen or preferably hydrogen chloride. Between 10% and 50% by volume of hydrogen chloride is desirable. However, as hydrogen chloride is formed as by-product in the substitutive chlorination of hydrocarbons it may not be necessary to provide an external source of hydrogen chloride in a recycle system. The hydrogen chloride can be built up to the desired level by recycling the reactor exit gases after removal of the chlorinated products.

Unpacked Columns

The chlorination reaction should preferably be carried out in unpacked columns with the reaction temperature controlled to between 330° and 420°C for a residence time of not more than 12 seconds and at atmospheric pressure. It is preferred to carry the vapour phase reaction continuously in an empty tube in the absence of a catalyst. The reaction may be adiabatic or with heat removal.

Distillation or extraction methods may be used to work up the reaction products. Hydrogen chloride may be separated from the head products by any convenient means such as water-scrubbing and the hydrocarbons recycled, preferably after drying; or the reactor products may be absorbed in a scrubbing system, leaving gaseous dry hydrogen chloride as a valuable by-product.

Products of the reaction consist mainly of 3:4-dichlorobutene-1 and 1:4-dichlorobutene-2 from the butadiene, and of monochloro-substituted derivatives of the alkane and alkenes. These products are, of course, valuable intermediates in the preparation of chloroprene.

Examples illustrating the process are quoted.

In the patent on the preparation of 3:4-dichlorobutene-1 an improved process is given for preparing straight chain dichlorobutenes by chlorination of unsaturated C₄ hydrocarbons in the vapour phase, and is a development of the process described in British Patent 798,393 (see above).

It is stated that the presence of even small amounts of dichlorobutanes in the chlorination reaction is disadvantageous. These dichlorobutanes, formed from butene-1 or butene-2, have boiling points very close to that of 3:4-dichlorobutene-1. As a result separation of 3:4-di-

chlorobutene-1 uncontaminated by dichlorobutanes by fractionation of the reaction product is very difficult. If the 3:4-dichlorobutene-1 is subsequently converted by dehydrochlorination into chloroprene (British Patent 798,205) dichlorobutanes present will be partly converted into mono-chlorobutenes which boil within a degree or two of the chloroprene, with the result that the purification of the chloroprene is rendered difficult.

In this patent therefore a process is suggested for the production of 3:4-dichlorobutene-1 from butadiene-1:3 containing n-butene. The butadiene is reacted in the vapour phase at an elevated temperature with chlorine to produce a reaction product containing dichlorobutenes contaminated with dichlorobutanes. This reaction product is treated, preferably after separation of the 1:4-dichlorobutene-2, to isomerise the 3:4-dichlorobutene-1 into 1:4-dichlorobutene-2, and then separating the 1:4-dichlorobutene-2 from the isomerisation product by distillation, and reconverting the 1:4-dichlorobutene-2 into 3:4-dichlorobutene-1.

"An important feature of the process of the present invention is that butadiene containing any proportion of n-butene can be used and such a mixture is likely to be cheaper and more readily available than pure butadiene."

Isomerisation

Isomerisation may be carried out by heating the chlorination product in the absence of a catalyst but it is claimed that isomerisation is preferably carried out by heating the chlorination product in the presence of a metal halide condensation catalyst at a high temperature. A wide range of catalysts is available and examples of these are given in the patent. In particular a cuprous or cupric salt in combination with an organic amine as described in British Patent 798,889 may be used. It is stated that the amounts of catalyst used in the isomerisation are not critical. "In general amounts between 0.1 and 1.0% by weight of the copper chloride and an amount of amine between about 2 and 5 times the weight of copper chloride."

The isomerisation step converts the dichlorobutenes into an equilibrium mixture of 1:4-dichlorobutene-2 and 3:4-dichlorobutene-1. When a substantial amount of the 1:4-isomer has been formed, the catalyst is removed by flash distillation or by washing with hydrochloric acid, and the mixture is distilled to separate the lower boiling constituents including the dichlorobutanes and residual 3:4-dichlorobutene-1 from the higher boiling 1:4-dichlorobutene-2. The head product may be isomerised to equilibrium again and redistilled.

Recovered 1:4-dichlorobutene-2, free from dichlorobutanes is reconverted into 3:4-dichlorobutene-1 in a second isomerisation step similar to the first. Since the desired isomer is the lower boiling of the two, isomerisation "can be readily carried to completion by continually distilling the reaction mixture in the presence of the isomerisation catalyst."

Instrument Makers and Users Should Work Together



This photograph taken at the BIMCAM luncheon in London shows, l. to r.: Sir Arnold Hall, president Royal Aeronautical Society, Sir Ewart Smith, a deputy chairman of I.C.I., and L. S. Yoxall, president, BIMCAM

INSTRUMENTATION was of growing importance in the process industries. Sir Ewart Smith, a deputy chairman of I.C.I., told members of the British Industrial Measuring and Control Apparatus Manufacturers' Association.

Proposing the toast of the association at their annual luncheon in London, he said that in the old days industrial plants were designed first and instrumentation was added as something of an after-

thought: today it was an integral part of the design.

Sir Ewart recommended an ever closer association between instrument manufacturers and users so that an even greater understanding of each other's problems might be achieved.

Replying, Mr. D. B. Pinkney, chairman of BIMCAM, said that what the instrument manufacturers were really selling was an engineering service.

U.S. Development Simplifies a Key Step in T.E.L. Manufacture

A NOVEL measuring instrument developed by Stanford Research Institute, Menlo Park, Cal., U.S., is stated to simplify a key step in manufacturing tetraethyl lead (T.E.L.). It permits continuous analysis and exceptionally close control of the composition of a sodium-lead alloy used in making the antiknock preparation. Precision is important as the amount of sodium in the alloy is critical in order to obtain a maximum yield of T.E.L. from the chemical manufacturing process.

Previously, control of sodium content required laboratory analysis of samples taken at intervals and adjustment of the alloy composition according to the analytical results. The Ethyl Corporation of America desired therefore a method of monitoring the sodium content which would allow for continuous analysis of the sodium-lead alloy. A variation of $0.01\% \pm$ in the sodium present was specified as the maximum permissible. The instrument was also required to operate at temperatures up to 930°F .

The system, developed and further refined for industrial application by C. I. Glassbrook, is based, essentially, upon measuring the ratio between pure sodium and the varying concentration of sodium in the alloy. A glass tube containing pure sodium is immersed in the batch of molten sodium-lead alloy and connected by an electrical lead to a recording potentiometer. Another electrical lead is inserted directly into the alloy. The difference in potential between the pure sodium and the alloy is measured by the potentiometer which is calibrated in terms of the percentage of sodium in the alloy.

This sodium analyser comprises a

sodium content recorder, temperature recorder, and a controller enabling content recorder to measure the sodium content to an accuracy of 1 part in 4,000. Refined for industrial application, this analyser is now in current use at all Ethyl Corporation T.E.L. production plants.

Work Begun on New British Standards

WORK has been started by the British Standards Institution on a number of new standards covering the following: rigid p.v.c. extrusion and moulding compounds; unsaturated polyester resins for low-pressure fibre-reinforced plastics; lithium-based greases.

In addition draft standard CZ 2010 'Portable fire extinguishers of the carbon tetrachloride and chlorobromomethane types' has been circulated to industry for comment.

Among draft I.S.O. recommendations under consideration are the following: No. 187 'Detection of free ammonia in phenol formaldehyde mouldings (qualitative method)'; No. 188 'Determination of the percentage of styrene in polystyrene with Wijs solution'; No. 189 'Determination of viscosity number of p.v.c. resin in solution'; No. 190 'Method of test for determining the resistance of plastics to chemical substances'; No. 191 'Method of test for determining the loss of plasticiser from plastics by the activated carbon method'; No. 192 'Method of test for determining migration of plasticisers from plastics'; Nos. 243 to 256 on chemical analysis of manganese ores.

U.K. Output of Copper Sulphate Nearly Halved

GROSS output of copper sulphate for the first 11 months of 1958 totalled 24,694 long tons (41,090 long tons in the same period of 1957), according to the British Bureau of Non-ferrous Metal Statistics.

In the same 11 months U.K. consumption of lead included the following figures:

	Jan.-Nov. 1957	Jan.-Nov. 1958
	Long Tons	Long Tons
Battery oxides	22,859	24,286
Tetraethyl lead	19,394	18,178
Other oxides and compounds	22,665	23,631
White lead	6,885	6,278
Total all uses	322,626	308,766

U.K. consumption of tin for the period to 11 November 1958 by the U.K. chemical industry (mainly in the form of tin oxide) was 916 long tons (994 in the same period of 1957).

U.K. consumption of cadmium in the 11 months period rose from 878.60 long tons in 1957 to 931.85 long tons in 1958 as follows:

	Jan.-Nov. 1957	Jan.-Nov. 1958
	Long Tons	Long Tons
Plating anodes	466.60	465.80
Plating salts	70.65	72.45
Cadmium copper	40.75	41.30
Other alloys	31.40	31.40
Batteries: Alkaline	54.35	58.80
Dry	3.50	3.70
Solder	34.20	47.10
Colours	160.45	191.55
Miscellaneous uses	16.70	19.75
Total consumption	878.60	931.85

Consumption of antimony metal and compounds (in terms of antimony metal) was as follows:

	Jan.-Nov. 1957	Jan.-Nov. 1958
	Long Tons	Long Tons
Batteries	955	1,118
Other antimonial lead	417	545
Bearings	366	325
Miscellaneous uses	141	181
Oxides: for white pigments	1,467	1,211
For other uses	864	914
Sulphides (inc. crude)	56	59
Total consumption of antimony metal and compounds	4,266	4,353

New Research Company for Griffin and George

A NEW company, Griffin and George (Research and Development) Ltd., has been formed by Griffin and George Ltd., Alperton, Middx., to conduct research into and the development of new and scientific instruments and apparatus for laboratory use and process control.

New laboratories to house the research and development staff will be designed and equipped. A major part of the company's efforts will be directed towards a review of modern analytical techniques and the development of the instruments and apparatus for applying them in the laboratory and on the plant. At the same time, in view of its importance in educating the scientist of the future, work will be conducted on modernising and extending the group's range of apparatus for use in school laboratories, technical colleges and universities.

Research director of the new company is Dr. A. J. P. Martin, F.R.S. (see p. 212).

Overseas News

U.S. PRICE REDUCTIONS FOR L-P POLYTHENE EXPECTED TO ASSIST MARKET PROSPECTS

CUTS in the price of low-pressure polythene in the U.S., it is reported, should result in better market prospects. Phillips Chemical Co. have again led the cuts (as they did last year when the price was dropped to 43 cents per lb.). The present price is down to 38 cents per lb., nearly a 12% cut. Nearly all other U.S. producers have followed suit.

W. R. Grace say that the move is an effort to make "an orderly price transition" rather than the unpublished price reductions which have taken place in recent months. U.S. producers hope that the drop in price will increase usage of low pressure polythene. It is expected to lead to stiffer competition, however, for conventional polythene, present selling price of which is 35 cents per lb. A price cut for polypropylene has also been announced by Hercules Powder Co., who have reduced the price of their product by 7 cents per lb. The new price is 42 cents per lb., a drop of nearly 15%, and the third major reduction in just under a year. The natural colour moulding powder cost 65 cents per lb. in May last year.

Prices for Teflon fluorocarbon resins have been reduced by E.I. Du Pont de Nemours and Co., by 8 to 10% for all grades, effective as from 7 February. Price for the minimum quantity (truck loads) will now be \$4.10 per lb. Granular moulding powders are being cut 40 cents per lb., aqueous dispersions 45 cents and extrusion powder 80 cents. Reason for the price cuts here are stated by Du Pont to be due to a "major breakthrough in manufacturing technology."

Reduced prices for piperazine, piperazine hexahydrate and piperazine salts have also been announced by Jefferson Chemical Co., Houston, Texas. Anhydrous grade is \$1.60 per lb. (truckload quantity) and \$1.65 per lb. if under truckload quantity. Again, the price reduction is due to improved manufacturing techniques and also to a widening market.

Polythene Consumption in Finland

Consumption of polythene in Finland has risen from 48 tons in 1953 to 1,048 tons for 1957. In Sweden, consumption of polythene was about 400 tons in 1953 and 5,700 tons in 1957.

Professor G. Nyman, Teknillinen Korkeakoulu, Helsinki, estimates that polythene consumption in Finland is likely to rise a great deal, but due to the relatively small population of the country, will apparently stay under an economic production minimum which for low-pressure (Ziegler-type) polythene plant, has been given as 12,000 tons a

year. In the northern countries the matter of producing polythene, primarily by the high-pressure method, has been thoroughly studied, with the result, it is stated, that a somewhat lower production minimum will still be economical.

Ethylene may be manufactured from sulphuric acid or by cracking refinery gases. In the first-named instance, the price of the ethanol will determine the price of the ethylene; in the latter case production output will be a decisive factor in its price.

W. German Output of Chlorine in 1957

Figures relating to the West German production of certain chemicals have just been released. Output of chlorine in the year 1957, almost 520,000 tonnes, was 12% up on 1956. Imports for the year totalled 3,400 tonnes. Manufacture of chlorinated hydrocarbons took up most of the chlorine produced.

In the field of caustic alkalis the production of sodium hydroxide rose 9% on 1956 totals to 648,000 tonnes. Sodium hydroxide exports increased by 43% in 1957 to about 60,000 tonnes.

Canadian Expansion Includes \$910,000 Butadiene Plant

A new butadiene plant costing \$910,000 is provided for in a budget of \$6,660,000 for the Crown-owned Polymer Corporation at Sarnia, Ontario, tabled in the Canadian House of Commons. The figure for the plant is included in \$3,820,000 earmarked for new projects, including \$1,675,000 for improvements to production units.

Russia to get French Chemical Plant and Equipment

France, under a trade agreement extending to 1962, will sell to Russia enamelled equipment and spray driers for detergents this year. In due course, France will export a 20,000-tons-a-year titanium dioxide plant, a caustic soda plant (120,000-metric-tons-a-year capacity), polythene extrusion equipment worth \$2.8 million and miscellaneous chemical and plastics equipment to the value of \$20 million. Exchange of chemicals will continue. In exchange for coal-tar pitch, Russia will obtain French dyestuffs, pharmaceuticals, essential oils and other chemicals.

Rio Tinto Dow's Thorium Plant Well Advanced

Construction of the thorium extraction plant at Algoma Quirke, Ontario, Canada, started last summer by Rio Tinto Dow is reported to be well advanced. Work

is now continuing on the installation of plant process and control equipment.

The plant will be in operation by early spring, Mr. R. Winters, president of the company, reports. The \$1 million project, which is a joint undertaking by Dow Chemical Co. and Rio Tinto Mining Co., will eventually produce 100 to 200 tons of thorium salts annually. This is approximately equal to world current production of thorium in this form.

Another Shell Chemical Plant for Australia

Following the recent opening of Shell's sulphuric acid plant at Geelong, Victoria, Australia, and their decision to build an Epikote resin plant at Clyde, Sydney, Shell Chemical (Australia) Proprietary Ltd. are now to build a detergent alkylate plant at Geelong.

This plant, which will cost over A£1 million, is due for completion in early 1961. The production from the plant will, at full operation, represent an annual saving in foreign currency of over A£500,000.

Gas Pipeline Proposed from Russia to Sweden

Talks are proceeding on the subject of a natural gas pipeline from the U.S.S.R. to the east coast of Sweden. It has for some time been the aim of Russia to supply its natural gas and mineral oil resources to Scandinavia, particularly the Ukrainian deposits of natural gas. The projected pipeline would run from the Ukraine via the Baltic town of Memel and under the Baltic Sea.

Acrylonitrile Plant For Hungary

Production in the Hungarian plastics industry (about 5,000 tonnes in 1957) is to be increased to 12,000 tonnes a year by 1960. Basis for the production of p.v.c. and acrylonitrile will be natural gas piped from Rumania.

Ketones from Hüls

Small-scale production of the following ketones has been started by Chemische Werke Hüls, Marl, West Germany; all of them are colourless, immiscible with water and soluble in all organic solvents: 3-isopropyl-pentanone-2, K_{p10} 155-157°C; 3-isopropyl-heptanone-2, K_{p15} 78-79°C; 2-methyl-7-ethyl-nona-4, K_{p8} 89-90°C; 2-methyl-7-ethyl-decanone-4, K_{p10} 110-112°C. Other details have not yet been announced.

U.S. Antibiotic Sales

Chas. Pfizer's president, John E. McKeen, recently stated that the total value of antibiotics sold by the U.S. pharmaceutical industry for human medication, was estimated at \$380 million for 1958. Feed supplements, veterinary drugs, pesticides, preservatives and other

miscellaneous products took another \$70 millions' worth.

McKeen expects antibiotic sales to continue to rise due to a growing population and research discoveries. By 1965 he anticipates sales for human use will be valued at \$440 million, a 15% increase. Also more antibiotics will be used in agriculture and industry.

This year U.S. pharmaceutical industry, it is suggested, will spend about \$190 million for research. Pharmaceutical development abroad is also seen as spurring the U.S. drug industry to further developments.

Japanese Plans for Synthetic Rubbers

The Japanese company, Japanese Geon Co., are to start production of nitrile butadiene rubber and latex and high styrene rubber in July of this year. Initial production is to be 230 tons monthly. This will be raised by stages to 8,400 tons per year and then to 15,000 tons per year. In 1960 the Japan Synthetic Rubber Co. will also start production of rubbers.

Liquid Ammonia Production in Germany

The nitrogen plant of the Salzgitter iron and steel concern Hüttenwerk Salzgitter A.G. has started the production of 100,000 tonnes of liquid ammonia since its coming into operation late in 1956. The plant has a capacity of 48,000 tonnes of primary nitrogen annually. The ammonia is manufactured from gases produced by the coke ovens and furnace mouths from the Salzgitter foundry by the Haber-Bosch process. From the plant it is pumped to the premises of the subsidiary company Nordchemie, in Langelsheim, where it is processed into materials for synthetic fertilisers.

Canadian Sulphur Plant Suspends Operations

Operations are being suspended indefinitely next week by Noranda Mines at their multi-million dollar pyrites processing plant at Port Robinson, near Niagara Falls.

Built in 1954, the plant was designed to utilise the large deposit of low-grade pyrite at the company's Horne Mine, at Noranda, Quebec, for production of elemental sulphur, sulphur dioxide and high-grade iron oxide.

Reason for the closure is the change in the outlook for sulphur. When the plant was built sulphur was scarce. Now the new Mexican sulphur domes and recovery of sulphur from sour gas have changed the situation.

Lower U.S. Titanium Output

Production of titanium sponge in the U.S. dropped to about 10% of the industry's capacity by the middle of 1958 due to cutbacks in military requirements. In the third quarter, however, output began to increase. Total production for 1958 was about 4,500 short tons—one-fourth of the record 17,000 tons in 1957.

In the case of titanium mill, shipments were over 400,000 lb. a month in 1958, and for the year, 2,500 tons, about 50% of the 1957 figure. Producers expect that with gradual market diversification due to product development, usage will increase—particularly in the chemical industry. Titanium Metals Corporation consider that use of titanium will be confined to replacement of equipment because of corrosion problems, rather than preliminary designs based on titanium's properties, as in processes involving wet chlorine gas, metal chlorides, inorganic chloride salts and oxidising organic chlorinations.

Decarbonisation Compound from West Germany

The Ingelheim-on-Rhine company, Haus-Chemikalien G.m.b.H., has marketed a preparation for the decarbonisation of oil ovens. Applied to the inside of an oven when cold using an aerosol method of application, the solution, most of which consists of non-inflammable chlorine-fluorine hydrocarbons, produces

a covering skin on the carbon black formed there. This skin decreases the carbon's glowing temperature to 375-400°C and its effect is that on heating the carbon deposit 'glows-out'.

Canadian Company Buys Land for Plant

The Rohm and Haas Chemical Company of Canada, manufacturers of insecticides, fungicides, resins and other products, have bought 210 acres at Morrisburg, Ontario, for a new plant. The plant is expected to cost \$2 million (about £700,000).

U.S. Import Control

A new regulation has been issued by the U.S. Office of Civil and Defence Mobilisation which broadens its authority to restrict any imports whose volume imperils national security.

Fresh demands for tariff protection from domestic industry are expected to follow. Applications have been accepted in advance from the cobalt, tungsten and fluorspar industries.

Goodyear Report on Natsyn (Polyisoprene) Process and Pilot Plant

PILOT plant batches of *cis*-1,4-polyisoprene, a synthetic natural-type rubber developed by Goodyear Tire and Rubber Co., Akron, Ohio, and given the trade name Natsyn, have been produced for just over a year. The plant was set up to produce the material for product evaluation, and also to obtain data from which a commercial scale plant could be designed.

It has been found in practice that the process has its problems. Raw materials must be of high purity to avoid the presence of oxygen and some unsaturated compounds which would poison the catalyst. Even traces of water spoil the reaction. Towards the end of the reaction the high viscosity of the mixture of polyisoprene and solvent is such that heat transfer and agitation are difficult. Throughout the process also, there must be careful control of conditions in order to obtain the most desirable polymer structure and molecular weight.

Raw Materials. Petroleum-derived isoprene is used and distilled before use as is the solvent used. Any straight-chain hydrocarbon can be employed but the preferred solvent is *n*-pentane which has a boiling point close to that of isoprene. Isoprene and solvent are measured by weight, mixed and dried in a silica gel or alumina dehydrating unit.

Equipment. The plant is composed of four stainless steel reactors which are identical except for size and which can be interchanged. For examining new process conditions two 26 gall. reactors are used. The larger, 500 gall. reactors, are used to produce Natsyn for test tyres. On these latter reactors turbine-type mixers driven by 10 h.p. motors are employed.

Process. The process is described as a straightforward batch reaction (*Industrial*

and Engineering Chemistry

January issue). Isoprene monomer is mixed with the hydrocarbon solvent, the catalyst (a Ziegler catalyst is preferred by Goodyear but the plant is designed to use either Ziegler-type or lithium-derived catalyst) is added and the mixture is reacted at a moderate temperature (50°C is a good average) and pressure. The solvent is removed.

The reaction is stated to start as soon as the catalyst is added.

Heat from the exothermic polymerisation is removed by

water or brine circulating in the jacket of the reactor.

The temperature of 50°C is a good average temperature.

A lower temperature gives polymers of high molecular weight, but catalyst consumption increases and the reaction rate is slower.

Every two hours the cement's solids content is checked. At 7% solids the mixture is viscous and although thixotropic, is reported as still difficult to cool. Actual viscosity during polymerisation has not been measured, but a laboratory test has shown a 15% solids sample to have 100,000 centipoises. With experience, Goodyear are now able to operate the plant at 25% solids and they hope to push this figure even higher. Higher solids content increases the amount of polymer per batch and cuts down the quantity of solvent that must be stripped off.

From the reactor the batch is pumped to a holding tank where catalyst deactivator and antioxidant are added. Solvent is removed in an extruder, since this offers a convenient method of obtaining a closed system for good solvent recovery. The vapoured solvent is condensed, distilled and is then ready for re-use.

Aim of Goodyear is to develop a more efficient process and one that is continuous.

Liquid Oxygen Storage For New Lurgi Gas Plant at Westfields

LARGE quantities of oxygen in reserve supply will be possible with the installation of Britain's first Lurgi pressure-gasification plant which the Scottish Gas Board is building at Westfield, Fifeshire. The plant will make gas by reacting low grade coal with oxygen and steam under pressure.

As it is essential in any gasworks to ensure continuity of production, because of continuous consumer demand, it was decided that large quantities of oxygen should be kept in reserve at Westfield to meet this requirement without installing unnecessary stand-by plant. Oxygen will be stored in liquid form and the necessary equipment, operating on the Tonnox internal compression system, is being built by British Oxygen Engineering Ltd.

The advantage of the Tonnox system is that a comparatively large amount of oxygen can be drawn off for storage in liquid form when necessary and the actual proportion available in liquid form can be varied within wide limits. Storage capacity at Westfield will accommodate 550 tons of liquid oxygen.

The £750,000 contract for the tonnage oxygen plant was recently awarded to British Oxygen Linde Ltd. by the main contractors, Humphreys and Glasgow Ltd. The oxygen plant which will be entirely of British design and manufacture, is being built by British Oxygen Engineering. The plant will be capable of producing 200 tons of oxygen a day and is scheduled to go into production in the latter half of 1960.

New Cement Resists Both Acids and Alkalies

A NEW cement, Alcuma, will, it is claimed, permanently withstand the action of both acids and alkalies. The cement, made by J. H. Sankey and Son Ltd., Ilford, Essex, is composed of polyester resins combined with high quality inert fillers to produce an impermeable cement. It comes to the user in the form of a powder and solution, supplied separately.

It is claimed that this cement is eminently satisfactory for bedding and jointing, or pointing, vitrified tiles and bricks in the construction of acid and alkali resisting floors, benches, tanks, brickwork and so on. It can also be used for jointing and sealing pipes carrying acids and alkalies.

Need For New Gas Processes

At a House of Commons meeting of the Parliamentary and Scientific Committee, Mr. A. H. A. Wynn, scientific member of the National Coal Board, stated that investment in new coal carbonising plant in the gas industry was apparently coming to an end. He said that a large number of new plants were being built to make town gas from oil.

The policies of the gas industry were not the N.C.B.'s responsibility, but from the board's point of view, new processes were needed that were more economic than carbonisation.

U.K. Develops Europe's Largest Radiation Absorbing Glasses

DEVELOPMENTS in nuclear research in recent years have meant an increased demand for viewing windows to permit clear observation of operations or objects within the bounds of the 'hot' cell walls, and to facilitate the remote handling of radioactive materials, while providing protection for the operatives.

Just as ingenious mechanisms have been evolved for remote handling, so have special types of glass been produced for shielding windows. In the forefront in this field is the Chance-Pilkington Optical Works at St. Asaph, North Wales, which has now developed radiation absorbing glasses of larger dimensions than have been available hitherto in Europe.

Particularly at very short wavelengths, the attenuation offered by protective walling is proportional to the density of the material employed. The Chance-Pilkington shielding glasses range in density from 2.5 g. per cc. to 6.1 g. per cc., and allow the design of windows to be, in many cases, of equal thickness to the surrounding wall, thereby reducing the complexity of additive shielding and obtaining the maximum optical advantage from the window.

On receipt of large dosages of short wavelength radiation, glasses of normal composition tend to discolour to a deep yellow or brown. To overcome this, Chance-Pilkington manufacture a series of glasses which are stabilised. These glasses have amended compositions having the effect of slightly decreasing the transmission of the glass in the un-irradiated state, but reducing the tendency for further discolouration under conditions of radiation.

Shielding glasses of density 2.5 g. per cc. and 4.3 g. per cc. are manufactured in polished plate form and also in block

form up to 4 ft. 6 in. by 3 ft. in area and 10 in. in thickness, with the limitation for the time being that no single block shall weigh more than 2,000 lb.

The Chance-Pilkington design and advisory service will consider specific requirements and design complete composite glass shielding windows, fully framed. These windows will thus be despatched in a form which will permit immediate and simple installation into the shielding wall on arrival at site, and if necessary technical staff can supervise installation.

New Laboratory For Cambridge Instrument

CONSTRUCTION of a new research laboratory has started for the Cambridge Instrument Co. Overlooking the River Cam and Jesus Green, and within ten minutes' walk of the centre of Cambridge, the 20,000 sq. ft. building will provide an imposing frontage for the factory.

The top floor with generous roof lighting will form an ideal design and drawing office, and the three floors below are being fitted with the latest equipment for instrument work involving physics, chemistry, electronics and precision mechanical engineering.

International Instrument Show

Products from over 50 factories from 10 countries will be shown at the 5th International Instrument Show, sponsored by B. and K. Laboratories Ltd. The exhibition will be held at the International Instrumentation Centre, 4 Tilney Street, Park Lane, London, from 6 to 10 April. Emphasis will be on new high speed analogue and digital instrumentation, etc.

New Pyrethrum Extract Plant

IN 1948 growers received from the Pyrethrum Board of Kenya a total of £165,000, a figure that in 1958 rose by £1,226,000. In the same period the board's capital assets rose from £71,000 to £208,000. These figures were given by Sir Evelyn Baring, Governor of Kenya, at a lunch held recently at Nakuru by

the board to mark the annual delegate conference of the growers.

During the conference, Mr. D. H. Pell Smith, chairman of the board, stated that cost of the newly built pyrethrum extract plant would be within the estimate of £200,000. The plant, shortly due to come into production, is at Nakuru.



D. H. Pell Smith, chairman, Pyrethrum Board of Kenya (centre), shows the new extract plant to two growers

NEW 'DOUBLE-ENDING' PROCESS AIDS PIPE WELDING ON SITE

TO make 'double-ending' welding of pipelines on site a speedier and more practical proposition, Quasi-Arc Ltd., Bilston, Staffs, have developed new techniques. The development follows the need for increased speed of pipeline welding resulting from expanding development programmes of the oil and chemical industries.

In 'double-ending' welding, pipe lengths are first automatically welded in pairs before being laid. This reduces the amount of manual welding on site by almost half.

A new manipulator has now been designed for improved handling of the pipe lengths. Driving rolls are used along the complete length of pipe, whereas the previous 'double-ending' machine used only main driving rolls, with idler rolls along the rest of its length. This new machine will handle pipes up to 40 ft. long in pairs and, to accommodate different diameters, the welding head is fully adjustable on the vertical column.

In operation these manipulators accept

the pairs of pipes on a cradle fitted with rollers, which, in much the same way as the roller beds used for large vessels, rotate the component while the welding equipment remains stationary. The handling of pipe lengths on and off the roller bed is carried out by hydraulic means. Speed of rotation is varied according to the pipe being welded and the automatic equipment and welding technique employed.

The new manipulator is designed to ensure correct and even speeds and to give consistent alignment under the welding head, without allowing the work to drift longitudinally along the rolls.

Any of the normal Quasi-Arc automatic welding processes, including Fusarc/CO₂, Fusarc or Unionmelt can be used with the new equipment. It is designed as a self-contained unit so that it can be used either in the shop or on site. Complete engine-driven equipment provides a.c. power-operation of the rollers and d.c. supply for welding and all ancillary supplies.

Britain's Biggest Carbon Monoxide Boiler

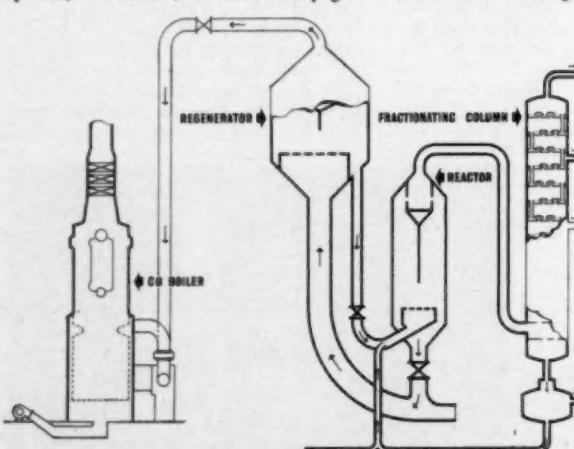
THE Fawley refinery of Esso Petroleum Co. Ltd., is to have Britain's largest carbon monoxide boiler, designed, manufactured and installed by Babcock and Wilcox Ltd. This boiler will use the flue gases, rich in carbon monoxide, produced during the catalytic cracking process, to generate 300,000 lb. of steam per hour at 150 p.s.i. and a temperature of 465°F.

It is of the circular, vertical type surrounded by a steel chimney, with an overall height of some 250 ft., and will be used primarily to supply steam for the catalytic cracking unit. The boiler is also equipped with combined Babcock Multispud gas burners and oil burners for employing a supplementary fuel supply of either fuel oil or refinery gas or both.

The oil burners are used for starting up and, combined with the refinery gas

burners, generate the initial supply of steam to the cracking unit. As the throughput of the cracking process increases the volume of carbon monoxide gas mixture increases, and the supplementary supply of fuel oil and/or refinery gas is reduced until a condition is reached when the ratio of supplementary fuel to carbon monoxide flue gas burned is in the proportion of one-third to two-thirds approximately, and under these conditions the boiler will operate at full capacity.

The Babcock carbon monoxide boiler is a self-contained unit which not only recovers and burns the sensible heat and heat of combustion in the carbon monoxide flue gases from the catalytic cracker but can, when using only the supplementary supply of fuel, maintain a full steam supply to the refinery even when the cracking unit is shut down.



This simplified diagram shows a Babcock and Wilcox CO boiler operating from the flue gas of a fluid-type catalytic cracking unit

DIARY DATES

MONDAY, 2 FEBRUARY

R.I.C.—Dartford: College of Technology, Miskin Rd., 7 p.m. 'Man-made fibres', by M. W. Alford.

S.C.I.—London: 14 Belgrave Sq., S.W.1, 6.30 p.m. 'Selection and control of semiconductors for different applications', by D. A. Wright.

TUESDAY, 3 FEBRUARY

I.Chem.E.—Manchester: Chem. Eng. Bldg., Jackson St., 3 p.m. Symposium on effluent disposal and air pollution.

Inst. Metals—Oxford: Cadena Cafe, 7 p.m. 'New metals: the rare earths', by A. D. McQuillan.

S.A.C.—London: School of Hygiene, 6.30 p.m. 'Study of distribution of X' in insulin assays', by J. V. Smart and G. A. Stewart; 'Use of range method in estimating variance in biological assays', by V. J. Birkinshaw; 'Comparison of approximate with more refined methods for treating 2-2 quantal response assays', by K. L. Smith.

S.C.I.—London: 14 Belgrave Sq., S.W.1, 6 p.m. 'Polymers and derivatives of phosphonitrile chloride', by F. R. Gimblett.

WEDNESDAY, 4 FEBRUARY

Inst. Fuel—London: Inst. Civil Eng., 1-7 Gt. George St., S.W.1, 5.30 p.m. 'A radioactive corrosion probe', by D. Anson.

R.I.C.—Walthamstow: College of Technology, Forest Rd., E.17, 7 p.m. 'Petroleum chemicals', by S. F. Birch.

S.A.C.—London: C.S., Burlington Hse., W.1, 7 p.m. Papers on 'Estimation of pesticide residues', by E. Q. Laws, J. G. Reynolds and M. G. Ashley.

S.C.I.—London: 14 Belgrave Sq., S.W.1, 6.15 p.m. 'Practical implications of the laboratory evaluation of protein quality', by G. D. Rosen.

THURSDAY, 5 FEBRUARY

C.S.—Bristol: Chemistry Dept., Univ., 5.15 p.m. Lecture by R. C. Cookson.

C.S. with R.I.C.—Sheffield: Chemistry Dept., Univ., 4.30 p.m. 'Ion-pairs in reaction kinetics', by C. W. Davies.

Plastics Inst. with Inst. Rubber Industry—Southampton: Chemistry Dept., Univ., 7.30 p.m. 'Vinyl chloride polymers: their design and application', by B. S. Dyer.

Polarographic Soc.—London: Duke of York, 8 Dering St., W.1, 7 p.m. 'Some polarographic problems encountered in semiconductor field', by V. J. Jennings.

R.I.C.—Kingston: Technical College, Fassett Rd., 6.30 p.m. 'Fluorinated hydrocarbons', by M. Stacey.

R.I.C. & S.C.I.—Leeds: Queen's Hotel, 7.30 p.m. 'Alcohol, antibiotics, cows and chemistry', by Magnus Pyke.

R.S.—London: Burlington Hse., W.1, 4.30 p.m. 'Exchange interactions in antiferromagnetic salts of iridium', by A. H. Cooke, et al.

S.C.I.—London: 14 Belgrave Sq., S.W.1, 6.15 p.m. Two papers on nitrogen fixation.

S.C.I.—Loughborough: Genatosan's Canteen, 7.30 p.m. 'Ladies' evening: 'Petrochemicals in the home'.

S.C.I.—Newcastle upon Tyne: King's College, 6.30 p.m. 'Recent research at Brewing Industry Research Foundation', by A. H. Cook.

FRIDAY, 6 FEBRUARY

C.S.—Birmingham: Chemistry Dept., Univ., 4.30 p.m. 'Birchage molecules', by F. G. Mann.

C.S.—Cambridge: Chemical Lab., Lensfield Rd., 8.30 p.m. 'X-ray analysis of complex structures', by D. Hodgkin.

C.S.—Exeter: Washington Singer Labs., Prince of Wales Rd., 5 p.m. Pedler lecture: 'Polyacetylenes', by E. R. H. Jones.

Royal Inst.—London: 21 Albemarle St., W.1, 9 p.m. 'Microscopy with X-rays', by V. E. Cosslett.

R.I.C.—Norwood: Technical College, Knight's Hill, S.E.27, 6.30 p.m. 'Films on glass, crystallisation and electron microscope'.

S.C.I.—Glasgow: Royal College, 7.15 p.m. Jubilee memorial lecture: 'Colonial research—products and pesticides', by R. A. E. Galley.

S.C.I.—Manchester: Robinson Lecture Theatre, Univ., 6.30 p.m. 'Chemical constitution and physical behaviour of waxes', by C. Jones.

Soc. Instrument Technology—Fawley: Cophthorne Hse. 'Laboratory and industrial pH measurements and titrations', by G. Mattock.

SATURDAY, 7 FEBRUARY

I.Chem.E.—Birmingham: College of Technology, Gosta Grn., 2.30 p.m. A.G.M. of Midlands branch: 'Principles of selecting dimensional systems and units in chemical engineering', by A. Klinkenberg.

Institution of Plant Engineers

With effect from 16 January, the Incorporated Plant Engineers has changed its name to the Institution of Plant Engineers.

UK Chemical Exports and Imports for 1957 and 1958

EXPORTS	QUANTITY		VALUE	
	1957	1958	1957	1958
INORGANIC				
Acids	Cwt.	211,930	181,090	£ 690,948
Copper sulphate	Tons	28,427	26,517	2,460,399
Sodium hydroxide	Cwt.	4,181,995	3,821,351	5,347,253
Sodium carbonate	"	4,260,469	3,723,344	2,917,910
Aluminium oxide	Tons	32,725	38,232	1,139,028
Aluminium sulphate	"	34,619	34,741	489,735
Other aluminium cpds.	"	3,323	3,348	154,910
Ammonia	Cwt.	78,785	90,148	169,898
Ammonium cpds. (not fertilisers or bromide)	Tons	18,520	19,192	722,442
Arsenical compounds	"	3,727	3,123	264,538
Bismuth compounds	Lb.	388,460	379,690	319,730
Bleaching powder (chloride of lime)	Cwt.	250,075	264,618	427,090
Hydrosulphite	"	86,127	63,860	672,400
Other bleaching materials	"	143,769	175,976	677,290
Calcium compounds	"	356,189	408,781	713,767
Carbon blacks	"	774,449	766,932	2,844,415
Cobalt compounds	"	16,950	19,003	590,214
Iron oxides, chemically manufactured	"	95,125	82,738	289,452
Lead compounds	"	59,067	76,328	477,238
Magnesium cpds. (n.e.s.)	Tons	17,603	16,455	876,609
Nickel salts	Cwt.	82,654	65,956	817,646
Potassium compounds (not fertilisers or bromides)	"	61,581	56,932	600,993
Sodium bicarbonate	"	726,550	674,160	667,423
Sodium phosphates	"	133,921	201,142	604,496
Sodium silicate (water glass)	"	323,728	344,578	290,325
All other sodium cpds.	"	1,771,787	1,723,461	4,00,973
Tin oxide	"	8,570	7,705	310,797
Zinc oxide	Tons	6,831	9,024	494,694
Inorganic chemical elements or compounds, inc. radioactive (n.e.s.)	"	—	—	4,765,688
ORGANIC				
Acids, anhydrides & their salts & esters	Cwt.	101,875	48,115	1,361,037
Glycerine	"	—	939,510	480,751
Ethyl alcohol, etc. & mixtures of alcohols (n.e.s.)	"	—	—	1,586,755
Acetone	Cwt.	130,003	182,643	410,232
Citric acid	"	49,586	45,556	490,897
Gases, compressed, liquid or solid (n.e.s.)	"	—	—	2,304,206
Phenol	Cwt.	138,399	168,196	881,953
Salicylates	Lb.	987,866	723,149	277,806
Sodium compounds	Cwt.	29,771	22,833	401,259
Sulphonamides not prepared	Lb.	1,520,451	1,901,992	1,138,127
Dyestuffs intermediates (n.e.s.)	Cwt.	98,847	83,301	1,381,165
Organic compounds, inc. radioactive (n.e.s.)	"	—	—	16,579,018
Total for elements & cpds.	"	—	—	62,646,636
Coal tar	Tons	89,250	72,308	971,896
Cresylic acid	Galls.	3,081,132	1,836,494	1,109,057
Creosote oil	"	18,461,902	16,721,211	639,607
Other mineral tars and crude chemicals	"	—	—	1,150,936
Pigment dyestuffs	Cwt.	29,605	27,682	458,037
Other syn. org. dyestuffs	"	187,851	163,692	9,112,777
Synthetic organic pigments	Cwt.	25,613	23,114	993,626
Vegetable & animal dyes	"	3,473	2,870	129,525
Tanning extracts (solid or liquid)	"	124,519	111,214	556,611
Synthetic tanning materials	"	78,802	79,110	301,002
Pigments, paints, varnishes, putty, etc.	"	—	—	24,178,442
Drugs, medicines, etc.	"	—	—	39,634,158
Antibiotics	"	—	—	8,758,580
Explosives	"	—	—	0,546,885
Detergents (syn. org.)	Cwt.	1,166,946	913,875	7,706,211
Soap substitutes	Gall.	5,465,346	5,269,962	1,929,524
Tetraethyl lead	"	79,526	64,652	337,993
Carbons	Cwt.	—	—	269,861
FERTILISERS, ETC.				
Ammonium nitrate	Tons	2,148	1,181	70,703
Ammonium sulphate	"	205,804	304,287	3,528,267
Other N fertilisers	"	—	—	469,248
Disinfectants	Cwt.	165,111	145,097	1,271,758
Insecticides, etc.	"	345,271	277,923	4,240,770
Weedkillers	"	87,775	55,785	979,919
MISCELLANEOUS				
Plastics materials	Cwt.	2,271,644	2,482,912	29,921,768
Photographic chemicals (n.e.s.)	"	39,998	38,915	689,960
Scientific instruments	"	—	41,733,727	682,085
Chemical glassware	Cwt.	15,791	15,704	839,947
Chemical & gas machinery	"	157,582	242,970	3,553,063

IMPORTS	QUANTITY		VALUE	
	1957	1958	1957	1958
INORGANIC				
Acids	Cwt.	59,531	69,433	185,754
Aluminium oxide	Tons	23,067	27,948	1,440,366
Silicon carbide	"	5,539	7,942	981,710
Arsenic trioxide	"	3,294	6,891	215,438
Boron, refined	Cwt.	514,540	474,355	1,018,868
Calcium carbide	"	1,291,439	1,486,023	2,485,320
Carbon blacks, channel	"	178,287	173,160	1,005,556
Other carbon black	"	93,549	97,273	362,537
Cobalt oxides	"	7,710	6,330	483,196
Iodine	Lb.	949,532	1,112,707	399,494
Mercury	"	1,380,252	1,459,408	1,526,337
Sodium, calcium, potassium, lithium	Cwt.	24,058	116	199,391
Potassium carbonate	"	107,622	103,443	353,305
Other potassium cpds. (not fertilisers)	"	94,483	78,271	369,202
Selenium	Lb.	14,877	127,529	695,275
Silicon	Tons	5,607	6,898	951,562
Sodium chloride	Cwt.	107,525	135,297	363,940
Other sodium cpds.	"	338,840	406,054	1,237,895
Inorganic chemicals (n.e.s.)	"	—	—	3,028,006
ORGANIC				
Acids, anhydrides, salts & esters	Cwt.	—	—	2,869,786
Glycerine	"	107,566	211,153	659,948
Menthol	Lb.	163,803	145,634	327,549
Naphtha, methyl alcohol, alcohols & mixtures	"	—	—	3,456,621
Turpentine	Gall.	850,846	971,217	220,298
Glycol ethers & esters	Lb.	6,977,746	10,319,002	1,176,664
Sodium compounds	Cwt.	133,509	94,715	1,416,587
Styrene	Gall.	1,628,466	2,447,421	1,175,601
Vinyl acetate	Tons	4,913	5,026	568,803
Dyestuff intermediates	Cwt.	28,095	16,462	970,922
Organic compounds (n.e.s.)	"	—	—	692,269
Titanium dioxide	Cwt.	123,948	2,603	12,027,819
Drugs, medicines	"	—	—	1,162,533
Plastics materials	"	666,027	826,779	7,823,895
Polyvinyl chloride	"	—	332,817	9,589,765
FERTILISERS				
manufactured—				
Basic slag	Tons	110,318	114,958	906,045
Potassium chloride	Cwt.	10,749,823	11,639,973	9,527,586
Potassium sulphate	"	312,835	322,864	306,876
Other fertilisers	"	—	—	1,847,117
MISCELLANEOUS				
Chromium ore	Tons	196,562	178,738	3,041,043
Tantalum & niobium ores & concentrates	"	447	474	312,454
Titanium ores	"	267,486	269,560	3,435,236
Iron pyrites	"	356,830	245,123	1,323,770
Sulphur	"	325,712	328,437	4,793,617
Chemical & gas machinery	Cwt.	34,307	25,953	1,415,682
EXPORTS OF ALL CHEMICALS TO PRINCIPAL MARKETS				
	1956	1957	1958	
Ghana	—	—	4,603,238	5,150,265
Nigeria	—	—	5,404,304	5,309,032
Union of South Africa	—	—	11,790,065	12,823,189
Rhodesia and Nyasaland	—	—	2,378,326	2,923,942
India	—	—	18,265,084	16,561,003
Pakistan	—	—	4,209,608	3,820,340
Singapore	—	—	4,654,770	4,114,198
Federation of Malaya	—	—	3,742,133	3,840,015
Ceylon	—	—	2,480,469	3,614,509
Hong Kong	—	—	3,587,470	3,425,528
Australia	—	—	17,614,339	22,333,513
New Zealand	—	—	7,282,165	23,452,161
Canada	—	—	8,291,969	8,519,436
Irish Republic	—	—	6,630,113	6,775,354
Finland	—	—	3,227,015	2,812,390
Sweden	—	—	6,700,049	7,361,585
Norway	—	—	3,672,868	4,216,626
Denmark	—	—	4,621,767	4,793,873
Western Germany	—	—	6,239,633	8,515,567
Netherlands	—	—	8,857,346	10,025,849
Belgium	—	—	6,083,781	6,584,979
France	—	—	7,745,331	8,581,984
Switzerland	—	—	3,206,630	3,224,886
Portugal	—	—	2,187,217	2,630,845
Italy	—	—	8,824,815	8,777,262
Netherlands Antilles	—	—	2,751,540	3,297,977
Egypt	—	—	2,926,230	154,853
Iraq	—	—	2,350,173	2,671,234
Iran	—	—	2,089,181	3,365,765
Burma	—	—	3,124,790	4,237,494
China	—	—	1,282,799	2,788,407
Japan	—	—	2,286,736	2,286,058
United States of America	—	—	8,638,504	7,386,783
Venezuela	—	—	2,004,230	2,457,540
Argentine Republic	—	—	3,320,771	5,441,864
Total for all countries	—	244,540,905	267,432,119	263,132,417

● New director of the National Institute for Research in Dairying, Reading University, from 1 August next is PROFESSOR R. G. BASKETT, Professor of Agricultural Chemistry, Belfast University, and head of the chemical and animal nutrition division of the Ministry of Agriculture for Northern Ireland.

● MR. C. McCORMICK has joined Ex-Sud of South American Minerals and Products Co. Ltd. as sales executive primarily responsible for the sale of imported chemicals. He was formerly with Tar Residuals Ltd.

● DR. G. H. GREEN, B.Sc., Ph.D., F.R.I.C., and MR. G. B. WILSON, B.Sc., A.R.I.C., have resigned from the staff of the British Leather Manufacturers' Research Association, at Egham, Surrey. Dr. Green, who was head of the microbiology staff is joining the research staff of Quaker Oats Ltd. Mr. Wilson is joining the staff of the Calder and Mersey Extract Co. Ltd.

● MR. ROBERT E. CRYOR, president of North American Asbestos Corporation of Chicago, American subsidiary of the Cape Asbestos Company Ltd., has been elected president of Cape Asbestos (Canada) Ltd. of Toronto, and Caposite Insulations Ltd. of Sarnia, Canadian subsidiaries of Cape Asbestos.

● MR. ALBERT GALE has resigned as director and secretary of Wilton Antiseptic Ltd., and has been appointed managing director of Diversey (U.K.) Ltd., and Deosan Ltd.



G. K. Hampshire (left), chairman of I.C.I. General Chemicals Division, who is to join the main board (C.A., 24 January, p. 178). Right: Dr. H. Rogan, works general manager of the A.E.A. Springfield Works (see p. 199 of this issue)

● MR. GORDON HELPS, joint managing director of Humphreys and Glasgow, Ltd., of London, chemical engineers and contractors, has retired, some twelve months short of normal retirement age, on account of ill health. He is 63. Mr. Helps, a graduate of Leeds, joined Humphreys and Glasgow, Ltd., in 1919. He was appointed to the board in 1939, and became joint managing director in 1954.

● PROFESSOR F. S. SPRING, Ph.D., D.Sc., Freeland Professor of Chemistry at the Royal College of Science and Technology, Glasgow, is joining the board of Laporte Industries Ltd. as director of re-

PEOPLE in the news

search. The Hon. F. A. Leathers is also joining the board. He is chairman of William Cory and Son Ltd., and a director of numerous other companies.

● MR. H. G. DAVEY, works general manager for the Atomic Energy Authority at Windscale and Calder Hall, has, after a period of ill health, been appointed personal adviser to the managing director of the authority. Mr. TOM TUOHY, deputy general manager, will succeed him.

● DR. MARIAN K. KOCOR, professor of chemistry at the School of Agriculture, in Wroclaw, Poland, is working in the Perkin Laboratory of the Chemistry Department of the University of Manchester. He received a Polish state fellowship to come to England and chose the Perkin Laboratory because of the important work being carried out there in the field of experimental biosynthesis. Dr. Kocor has studied in Zurich under the 1939 Nobel Prize winner Professor Ruzicka.

● MR. RICHARD JAMES MANN and MR. ROBERT McEWAN have been appointed area sales managers of International Synthetic Rubber Company Ltd. Mr. Mann, the northern area sales manager, was previously technical sales representative with Monsanto Chemicals Ltd., and I.C.I. Mr. McEwan, until recently a technical representative with British Geon, of the Distillers group of companies, has been appointed southern area sales manager.

● MR. H. WEST, Assoc.M.C.T., Hon.M.Sc., M.I.Mech.E., M.I.E.E., has been appointed assistant managing director of Metropolitan-Vickers Electrical Co. Ltd., and ceases to act as director of electrical engineering. Mr. West is also a director of Metropolitan-Vickers; Beyer Peacock Ltd.; Sunvic Controls Ltd.; and Isotope Developments Ltd.; and chairman of Nuclear Graphite Ltd.

● MR. A. O. R. JOHNSON, managing director of T. H. and J. Daniels Ltd., has joined the board of Prat-Daniels (Stroud) Ltd.

● SIR ALAN HITCHMAN, Permanent Secretary of the Ministry of Agriculture, has been appointed to succeed Mr. W. Strath as a full-time member of the United

Kingdom Atomic Energy Authority. He will have special responsibility for external relations and commercial policy.

● MR. J. M. STOREY, managing director of Dewrance and Co. Ltd. has been appointed vice-chairman of the Council of British Manufacturers of Petroleum Equipment. Mr. Storey, who was awarded the C.B.E. in the New Year's Honours List, was the leader of the council's economic, technical and goodwill mission to Canada in 1957.

● MR. J. C. H. MCENTEE, chairman of the Wilton Council of Imperial Chemical Industries Ltd., is to succeed Col. J. Peckston as president of the Tees-side and South-West Durham Chamber of Commerce.

● After being a member of the board of directors of Oxley Engineering Co. Ltd., gas, chemical and structural engineers, Clarence Road, Leeds, for nearly 25 years, MR. H. SAVILLE has resigned.

● MR. C. E. WALTERS has been appointed general sales manager covering all the products of Sunvic Controls Ltd. and in addition, is retaining responsibility for the domestic products division.

● DR. E. M. MORGAN has been appointed to the newly created post of research manager at the Parke-Davis Hounslow laboratories. The post will form a centre



Dr. E. M. Morgan,
Parke-Davis
research manager

for the administration of the chemical research, pharmacological research, production development and clinical investigation facilities. Dr. Morgan graduated as B.Pharm. from the University of Wales, is an associate of the Royal Institute of Chemistry and a Ph.D. of London University. He has been a member of the Parke-Davis chemical research staff since 1951. MR. S. G. DAVIS, Birmingham, has been appointed superintendent of production.

● DR. A. J. P. MARTIN, M.A., Ph.D., F.R.S., well known for his work on partition chromatography, acknowledged by the award in 1952 of the Nobel Prize for Chemistry (jointly with Dr. R. Synge) has been appointed research director of Griffin and George (Research and Development) Ltd., Alperton, Middlesex. This is a new Griffin and George Group company (see also p. 206).

● The American Chemical Society announces that PROFESSOR ROBERT L. BURWELL, Jr., Northwestern University, has been elected chairman of the society's Division of Physical Chemistry for 1959.

Commercial News

Benn Brothers

Directors of Benn Brothers Ltd., publishers of CHEMICAL AGE, recommend payment of a 5% (same) interim dividend on ordinary and 3% (same) on preference, both for the half-year ended 31 December and both less tax.

British Titan Products

Gambian Minerals, owned by British Titan Products, are to close. Head of B.T.P., the world's third largest manufacturer of titanium dioxide, Mr. George Beeby, says Gambian Minerals is the one black spot on the company's otherwise "pure-white record." The loss involved will be about £1,000,000.

Mr. Beeby reports, however, that use of titanium dioxide is still growing. Capacity of the Grimsby plant is being increased from 55,000 tons to 70,000 tons a year. There are similar plans for overseas. The Australian company is being expanded, a new plant is under construction in South Africa and plans are being considered for Canada.

Imperial Chemical Industries, consolidated Zinc and Goodlass Wall each have a 30% interest in British Titan Products.

Fisons

At an extraordinary meeting on Friday, 13 February, Fisons Ltd. will create 5 million £1 ordinary shares and effect a one-for-two scrip issue. The directors state that the balance of the new shares will not be distributed in a way that would substantially alter the control of the company without the consent of the stockholders.

Hickson and Welch (Holdings)

Hickson and Welch are paying a final dividend of 9½% on a capital increased to £1m by a three-for-seven scrip issue, giving the equivalent of 12 3/10th% for the year ended 30 September 1958. This is almost exactly equal to the previous year's equivalent of 12 1/4%. In addition a special interim of 2½% has been declared in respect of 1958-59. Group profits expanded from £506,325 to £547,223. Net profit is £260,624 compared with £252,114, of which £258,553 (£251,397) is attributable to the parent company.

Thomas Tilling

Thomas Tilling Ltd. and Commercial Plastics Ltd. are to join in a new company, Mondart Ltd., which will introduce a range of aerosols. The Commercial Plastics group includes Fablon Ltd., makers of self-adhesive plastics sheeting, and the Fablon sales director, Mr. R. T. Copp, has been appointed managing director of the new company.

A.K.U. Profits

Algemene Kunstzijde Unie, the Dutch chemical manufacturers, announce that figures for the first three quarters of last

- **B.T.P. to Close Gambian Subsidiary**
- **Fisons to Create 5 m. £1 Ordinary Shares**
- **Hickson and Welch Profit Up £40,000**
- **A.K.U. Profit will be Higher by £360,000**

year show that the profit for 1958 will be about 21.4 million guilders (about £2,150,000). This is compared with a profit for the previous year of 17.9 million guilders (rather more than £1,790,000).

Member company of A.K.U., the American E.N.K.A. Corporation, it is announced, is to take over William Brand and Co. in Willimantico, Connecticut. This company manufactures various kinds of plastics-coated wire and cable.

Chemstrand Corporation

Record earnings are announced by Chemstrand Corporation for the last half of 1958. Net income from group operations in 1958, after all charges and provision for income tax, was \$18,354,724, against \$18,813,380 in 1957. Net income of the company was \$19,190,793 and \$20,054,608 respectively.

Farbwerke Hoechst A.G.

Farbwerke Hoechst A.G. are increasing their issued share capital from DM.462 million (about £39 million) to DM.556 million (about £47 million). The company's supervisory board has announced that the new issue will be floated at 150% of nominal value. Stockholders will have the first option on the new issue on a one-for-five basis.

Petrochim, Antwerp

The Antwerp company of Société Chimique des Dérivés du Pétroles (Petrochim) has announced a double rise in capital—from 484 million frs (approx. £3,460,000) to 667,100,000 frs. (approx.

£4,765,000) and from there to 700 million frs. (approx. £5 million)—both of these rises having taken place together.

Dominion Tar

The Dominion Tar and Chemical Company have made an offer for all the assets of the Gypsum Lime and Alabastine Canada, in which they already have an interest.

Dow Chemical

Consolidated earnings per common share of the Dow Chemical Company for the quarter ended 30 November 1958 were 64 cents (57 cents), making 1.07 dollars (1.12 dollars) for the six months to the end of November.

INCREASES OF CAPITAL

BRITISH INSECTICIDES LTD., 11/12 Moscowl Road, London W.C.2. Increased by £19,900 beyond the registered capital of £100.

HENRY W. PEABODY (INDUSTRIAL) LTD., manufacturers of detergents, oils, chemicals, etc., 16 Byward Street, London E.C.3. Increased by £15,000 beyond the registered capital of £25,000.

LANCASHIRE CHEMICAL WORKS LTD., 40 Brazenose Street, Manchester 2. Increased by £9,000 beyond the registered capital of £6,000.

NEW COMPANY

M. Z. SANDERS (CHEMICALS) LTD. Cap. £100. Manufacturers of and dealers in minerals, ores, clays, chemicals, etc. Directors: M. Z. Sanders and J. Bustin. Reg. office: 24/6 Regent Street, London.

Market Reports

BETTER DEMAND IS MAINTAINED

LONDON The improved home demand for industrial chemicals has been maintained during the past week and contract commitments are being drawn against with fair regularity. Export trade is also keeping up to a good level with a fair volume of overseas inquiries, but buyers seek competitive quotations. Supplies generally are easy.

Prices have been steadily held in most sections of the market and the under-tone remains firm enough.

There has been little change in the position of agricultural chemicals although rather more interest has been shown in fertilisers.

Business in coal-tar products is quietly steady with no changes to record.

MANCHESTER Prices on the Manchester chemical market during the past week have maintained a steady to firm front. The textile and allied industries are still calling for reduced supplies,

but contract deliveries to most other industrial consumers have been on a reasonably satisfactory scale. Shipping business also keeps up fairly well and there have been some fresh inquiries. Demand for fertiliser materials has slightly improved. The leading tar products are in steady, but quiet demand.

GLASGOW There was quite a demand during the past week for industrial chemicals and the overall position on the Scottish market was one of considerable activity. Quantities both in regard to spot and contract requirements were well maintained together with some for forward requirements, particularly for the basic heavy chemicals.

Some price changes took place, but mostly prices remain firm. Although it is rather early in the season, some interest is now being shown in agricultural chemicals, mostly concerning forward deliveries.

BRITISH

GENERAL CHEMICALS

Acetic Acid. D/d in ret. barrels (tech. acid barrels free); in glass carboys, £8; demijohns, £12 extra. 80% tech., 10 tons, £97; 80% pure, 10 tons, £103; commercial glacial, 10 tons, £106.

Acetic Anhydride. Ton lots d/d, £128.

Alum. Ground, f.o.r. about £25.

MANCHESTER: Ground, £25.

Aluminium Sulphate. Ex-works, d/d, £15 10s to £18.

MANCHESTER: £16 to £18.

Ammonia, Anhydrous. Per lb., 1s 9d-2s 3d.

Ammonium Chloride. Per ton lot, in non-ret. pack, £33 2s 6d.

Ammonium Nitrate. D/d, 4-ton lots, £31.

Ammonium Persulphate. Per cwt., in 1-cwt. lots, d/d, £6 13s 6d; per ton, in min. 1-ton lots, d/d, £123 10s.

Ammonium Phosphate. Mono-and di-, ton lots, d/d, £106 and £97 10s.

Antimony Sulphide. Per lb., d/d UK in min. 1-ton lots: crimson, 4s 9d d/d to 5s 2d; golden, 3s 1d d/d per lb. to 4s 5d d/d.

Arsenic. Ex-store, £45 to £50.

Barium Carbonate. Precip., d/d, 4-ton lots, bag packing, £41.

Barium Chloride. 2-ton lots, £49.

Barium Sulphate [Dry Blane Fixe]. Precip. 2-ton lots, d/d, £43.

Bleaching Powder. Ret. casks, c.p. station, in 4-ton lots, £30 7s 6d.

Borax. Ton lots, in hessian sacks, c.p. Tech., anhydrous, £68; gran., £46; crystal, £48 10s; powder, £49 10s; extra fine powder, £50 10s; BP, gran., £55 10s; crystal, £57 10s; powder, £58 10s; extra fine powder, £59 10s. Most grades in 6-ply paper bags, £1 less.

Boric Acid. Ton lots, on hessian sacks, c.p. Tech., gran., £76 10s; crystal, £84 10s; powder, £82; extra fine powder £84; BP gran., £89 10s; crystal, £96 10s; powder, £94; extra fine powder, £96. Most grades in 6-ply paper bags, £1 less.

Calcium Chloride. Ton lots, in non-ret. pack; solid and flake, about £15.

Chlorine, Liquid. In ret. 16-17 cwt. drums d/d in 3-drum lots, £41.

Chromic Acid. Less 2½%, d/d UK, in 1-ton lots, per lb., 2s 2d.

Chromium Sulphate, Basic. Crystals, d/d, per lb., 8½d; per ton, £79 6s 8d.

Citric Acid. 1-cwt. lots, per cwt., £11 5s. 5 cwt. lots per cwt. £11; packed in polythene (jute- or paper-lined) 1 cwt. lots, per cwt. £10 17s; 5 cwt. lots per cwt. £10 12s.

Cobalt Oxide. Black, per lb., d/d, bulk quantities, 13s 2d.

Copper Carbonate. Per lb., 2s 3d.

Copper Sulphate. F.o.b., less 2% in 2-cwt. bags, £74.

Cream of Tartar. 100%, per cwt., about £11 12s.

Formaldehyde. In casks, d/d, £39 10s.

Formic Acid. 85%, in 4-ton lots, c.p., £91.

Glycerine. Chem. pure, double distilled 1,2627 s.g., per cwt., in 5-cwt. drums for annual purchases of over 5-ton lots and under 25 tons, £11 1s 6d. Refined technical grade industrial, 5s per cwt. less than chem. pure.

Hydrochloric Acid. Spot, per carboy, d/d (according to purity, strength and locality), about 12s.

Hydrofluoric Acid. 60%, per lb., about 1s 2d.

Hydrogen Peroxide. Carboys extra and ret. 27.5% wt., £119 0s 0d; 35% wt., d/d, £143.

Iodine. Resublimed BP, under 1 cwt., per lb., 14s 1d; for 1-cwt. lots, per lb., 13s 2d; 5 cwt., per lb., 12s 8d.

CHEMICAL PRICES

These prices are checked with the manufacturers, but in many cases there are variations according to quality, quantity, place of delivery, etc. Abbreviations: d/d, delivered; c.p., carriage paid; ret., returnable; non-ret. pack., non-returnable packaging; tech., technical; comm., commercial; gran., granular.

All prices per ton unless otherwise stated

Iodoform. Under 1 cwt., per lb., £1 2s 4d, for 1-cwt. lots, per lb., £1 8s 8d, 5 cwt., per lb., 21s 1d, crystals, 3s more.

Lactic Acid. Pale tech., 44% by wt., per lb., 14d; dark tech., 44% by wt., per lb., 9d; chem. quality, 44% by wt., per lb., 12d; 1-ton lots, ex-works, usual container terms.

Lead Acetate. White, about £154.

Lead Nitrate. 1-ton lots, about £135.

Lead, Red. Basis prices: Genuine dry red, £104 5s; orange lead, £116 5s. Ground in oil: red, £125 5s, orange, £137 5s.

Lead, White. Basis prices: Dry English in 5-cwt. casks, £116; Ground in oil: English, 1-cwt. lots, per cwt., 194s.

Lime Acetate. Brown, ton lots, d/d, £40; grey, 80-82%, ton lots, d/d, £45.

Litharge. In 5-ton lots, £106 5s.

Magnesite. Calcined, in bags, ex-works, about £21.

Magnesium Carbonate. Light, comm., d/d, 2-ton lots, £84 10s under 2 tons, £97.

Magnesium Chloride. Solid (ex-wharf), £17 10s.

Magnesium Oxide. Light, comm., d/d, under 1-ton lots, £245.

Magnesium Sulphate. Crystals, £16.

Mercuric Chloride. Tech. powder, per lb., for 5-cwt. lots, in 28-lb. parcels, £1 1s 9d; smaller quantities dearer.

Mercury Sulphide, Red. 5-cwt. lots in 28-lb. parcels, per lb., £1 10s. 6d.

Nickel Sulphate. D/d, buyers UK, nominal, £170.

Nitric Acid. 80% Tw., £35 2s.

Oxalic Acid. Home manufacture, min. 4-ton lots, in 5-cwt. casks, c.p., about £132.

Phosphoric Acid. Tech. (s.g. 1.700) ton lots, c.p., £100; BP (s.g. 1.750), ton lots, c.p., per lb., 1s 4d.

Potash, Caustic. Solid, 1-ton lots, £95 10s; liquid, £36 15s.

Potassium Carbonate. Calcined, 96/98%, 1-ton lots, ex-store, about £74 10s.

Potassium Chloride. Industrial, 96%, 1-ton lots, about £24.

Potassium Dichromate. Crystal and gran., per lb., in 5-cwt. to 1-ton lots, d/d UK, £s 2d.

Potassium Iodide. BP, under 1-cwt., per lb., 8s; per lb. for 1-cwt. lots, 7s 3d.

Potassium Nitrate. 4-ton lots, in non-ret. pack, c.p., £63 10s.

Potassium Permanganate. BP, 1-cwt. lots, per lb., 1s 11½d; 3-cwt. lots, per lb., 1s 10½d; 5-cwt. lots, per lb., 1s 10d; 1-ton lots, per lb., 1s 10d; 5-ton lots, per lb., 1s 9½d. Tech., 5-cwt. in 1-cwt. drums, per cwt., £9 15s 6d; 1-cwt. lots, £10 4s 6d.

Sal ammoniac. Ton lot, in non-ret. pack, £47 10s.

Salicylic Acid. MANCHESTER: Tech., d/d, per lb., 2s 5½d, 1-ton lots.

Soda Ash. 58% ex-depot or d/d, London station, 1-ton lots, about £17 3s.

Soda, Caustic. Solid 76/77%: spot, d/d 1-ton lots, £33 16s 6d.

Sodium Acetate. Comm. crystals, d/d, £91.

Sodium Bicarbonate. Ton lot, in non-ret. pack, £21 10s.

Sodium Bisulphite. Powder, 60/62%, d/d 2-ton lots for home trade, £46 2s 6d.

Sodium Carbonate Monohydrate. Ton lot, in non-ret. pack, c.p., £46.

Sodium Chlorate. 1-cwt. drums, c.p. station, in 4-ton lots, about £88 10s.

Sodium Cyanide. 96/98%, ton lot in 1-cwt. drums, £126.

Sodium Dichromate. Crystals, cake and powder, per lb., 1s. Net d/d UK, anhydrous, per lb., 1s 1½d. Net. del. d/d UK, 5-cwt. to 1-ton lots.

Sodium Fluoride. D/d, 1-ton lots and over, per cwt., £5; 1-cwt. lots, per cwt., £5 10s.

Sodium Hyposulphite. Pea crystals, £38; comm., 1-ton lots, c.p., £34 15s.

Sodium Iodide. BP, under 1 cwt., per lb., 13s; 1-cwt. lots, per lb., 12s 9d; 5 cwt., per lb., 12s 3d.

Sodium Metaphosphate [Calgon]. Flaked, paper sacks, £133.

Sodium Metasilicate. (Spot prices) D/d UK in 1-ton lots, 1-cwt. free paper bags, £29.

Sodium Nitrate. Chilean refined gran. over 98%, 6-ton lots, d/d c.p., per ton £29.

Sodium Nitrite. 4-ton lots, £32.

Sodium Perborate. (10%) in 1-cwt. free kegs, cwt. lots, £139 5s.

Sodium Percarbonate. 12½% available oxygen, in 1-cwt. kegs, £170 15s.

Sodium Phosphate. D/d, ton lots: disodium, crystalline, £40 10s, anhydrous, £88; tri-sodium, crystalline, £39 10s, anhydrous, £86.

Sodium Silicate. (Spot prices) 75-84% Tw. Lancs and Ches., 4-ton lots, d/d station in loaned drums, £12 10s; Dorset, Somerset and Devon, per ton extra, £3 5s; Scotland and S. Wales, extra, £2 17s 6d. Elsewhere in England, not Cornwall, extra, £1.

Sodium Sulphate [Desiccated Glauber's Salt]. D/d in bags, about £20.

Sodium Sulphate [Glauber's Salt]. D/d, up to £18 10s.

Sodium Sulphate [Salt Cake]. Unground, d/d station in bulk, £10.

MANCHESTER: d/d station, £10 10s.

Sodium Sulphide. Solid, 60/62%, spot, d/d, in drums in 1-ton lots, £36 2s 6d; broken, d/d, in drums in 1-ton lots, £37 2s 6d.

Sodium Sulphite. Anhydrous, £71 10s; comm., d/d station in bags, £27-£28 10s.

Sulphur. 4 tons or more, ground, according to fineness, £20-£22.

Sulphuric Acid. Net, naked at works, 168° Tw. according to quality, £10 10s-£11 12s 6d; 140° Tw., arsenic free, £8 7s 6d; 140° Tw., arsenious, £8 2s 6d.

Tartaric Acid. Per cwt.: 10 cwt. or more, £14 10s; 1 cwt., £14 15s.

Titanium Oxide. Standard grade comm., rutile structure, £178; standard grade comm., anatase structure, £163.

Zinc Oxide. Max. for 2-ton lots, d/d, white seal, £100 10s; green seal, £98; red seal, £95.

SOLVENTS AND PLASTICISERS

Acetone. All d/d. In 5-gal. drums, £128; in 10-gal. drums, £118; in 40-45 gal. drums, under 1 ton, £93; 1-5 tons, £90; 5-10 tons, £89; 10 tons and up, £88; in 400-gal. tank wagons, £85.

Butyl Acetate BSS. 10-ton lots, £173.

n-Butyl Alcohol BSS. 10 tons, in drums, d/d, £149.

sec-Butyl Alcohol. All d/d. In 5-gal. drums, £168; in 10-gal. drums, £158; in 40-45 gal. drums, under 1 ton, £133; 1-5 tons, £130; 5-10 tons, £129; 10 tons and up, £128; in 400-gal. tank wagons, £125.

tert-Butyl Alcohol. 5-gal. drums, £195 10s; 40/45-gal. drums: 1 ton, £175 10s; 1-5 tons, £174 10s; 5-10 tons, £173 10s; 10 tons and up, £172 10s.

Diacetone Alcohol. Small lots: 5-gal. drums, £185; 10-gal. drums, £175. 40/45-gal. drums: under 1 ton, £148; 1-5 tons, £147; 5-10 tons, £146; 10 tons and over, £145, in 400 gal. tank wagons, £142.

Diethyl Phthalate. In drums, 10 tons, d/d, per ton, £210; 45-gal. drums, d/d, 1-4 drums, £216.

Diethyl Phthalate. In drums, 10 tons, per ton, £187 10s; 45-gal. drums, d/d, 1-4 drums, £193 10s.

Dimethyl Phthalate. In drums, 10 tons, per ton, d/d, £179, 45-gal. drums, d/d, per ton £185.

Diocetyl Phthalate. In drums, 10 tons, d/d, per ton £284; 45-gal. drums, d/d, per ton, £290.

Ether BSS. 1-ton lots, drums extra, per lb., 1s 11d.

Ethyl Acetate. 10-ton lots, d/d, £145.

Ethyl Alcohol [PB 66 o.p.]. Over 300,000 p. gal. 4s 0½d; d/d in tankers, 2,500-10,000 p. gal. per p. gal., 4s 2½d. D/d in 40/45-gal. drums, p.p.g. extra, 1d.

Absolute alcohol (75.2 o.p.), p.p.g. extra, 5d.

Methanol. Pure synthetic, d/d, £43 15s.

Methylated Spirit. Industrial 66° o.p.: 500-gal. and up, d/d in tankers, per gal., 5s 10½d; 100-499 gal. in drums, d/d, per gal., 6s 3d-6s 5d. Pyridinised 66° o.p.: 500 gal. and up, in tankers, d/d, per gal., 6s 2d; 100-499 gal. in drums, d/d, per gal., 6s 6d-6s 8½d.

Methyl Ethyl Ketone. All d/d. In 5-gal. drums, £183; in 10-gal. drums, £173; in 40/45-gal. drums, under 1 ton, £148; 1-5 tons, £145; 5-10 tons, £144; 10 tons and up, £143; in 400-gal. tank wagons, £140.

Methyl isoButyl Carbinol. All d/d. In 5-gal. drums, £203; in 10-gal. drums, £193; 40-45 gal. drums, less than 1 ton, £168; 1-9 tons, £165; 10 tons and over, £163; in 400-gal. tank wagons, £160.

Methyl isoButyl Ketone. All d/d. In 5-gal. drums, £209; in 10-gal. drums, £199; in 40/45-gal. drums, under 1 ton, £174; 1-5 tons, £171; 5-10 tons, £170; 10 tons and up, £169; in 400-gal. tank wagons, £166.

isoPropyl Acetate. In drums, 10 tons, d/d, £137; 45-gal. drums, d/d, £143.

isoPropyl Alcohol. Small lots: 5-gal. drums, £118; 10-gal. drums, £108; 40/45-gal. drums: less than 1 ton, £83; 1-9 tons, £81; 10-50 tons, £80 10s; 50 tons and up, £80.

RUBBER CHEMICALS

Carbon Disulphide. According to quality, £61-£67.

Carbon Black. UKARB-327-7½d. per lb. ex-works, 3 ton lots, under 3 tons but not less than 1 ton 7½lb ex-works, ex-store, London and Manchester, 8½d per lb.

Carbon Tetrachloride. Ton lots, £83 15s. **India-Rubber Substitutes.** White, per lb., 1s 5½d to 1s 8d; dark, d/d, per lb., 1s 1½d-1s 5d.

Lithopone. 30%, about £56 10s.

Mineral Black. £7 10s-£10.

Sulphur Chloride. British, about £50.

Vegetable Lamp Black. 2-ton lots, £64 8s. **Vermilion.** Pale or deep, 7-lb. lots, per lb., 15s 6d.

COAL TAR PRODUCTS

Benzole. Per gal., min. 200 gal., d/d in bulk, 90's, 5s. 3d; pure, 5s 7d.

Carbolic Acid. Crystals, min. price, d/d bulk, per lb., 1s 4d; 40/50-gal. ret. drums extra, per lb., 1d. Crude, 60's, per gal., 8s 4d.

MANCHESTER: Crystals, d/d, per lb., 1s 4d-1s 7d; crude, naked, at works, 8s 5d.

Creosote. Home trade, per gal., according to quality, f.o.r. maker's works, 1s-1s 9d. **MANCHESTER:** Per gal., 1s 2d-1s 8d.

Cresylic Acid. Pale 99/100%, per gal., 6s 6d; 99.5/100%, per gal., 6s 8d. D/d UK in bulk: Pale ADF, per imperial gallon f.o.b. UK, from 7s 8d to 9s 3d; per US gallon, c.i.f. NY, 100 to 118.5 cents freight equalised.

Naphtha. Solvent, 90/160°, per gal., 5s. 1d; heavy, 90/190°, for bulk 1,000-gal. lots, d/d, per gal., 3s 11d. Drums extra; higher prices for smaller lots.

Naphthalene. Crude, 4-ton lots, in buyers' bags, nominal, according to m.p.: £19-£30; hot pressed, bulk, ex-works, £40; refined crystals, d/d min. 4-ton lots, £65-£66.

Pitch. Medium, soft, home trade, f.o.r. suppliers' works, £10 10s; export trade, f.o.b. suppliers' port, about £12.

Pyridine. 90/160°, per gal., 15s-17s 6d.

Toluol. Pure, per gal., 5s 2d; 90's, d/d, 2,000 gal. in bulk, per gal., 4s 11d. **MANCHESTER:** Pure, naked, per gal., 5s 6d.

Xylool. According to grade, in 1,000-gal. lots, d/d London area in bulk, per gal., 6s-6s 3d.

INTERMEDIATES AND DYES

(Prices Nominal)

m-Cresol. 98/100%. 10 cwt. lots d/d, per lb., 4s 9d.

o-Cresol. 30/31°C. D/d, per lb., 1s.

p-Cresol. 34/35°C. 10 cwt. lots d/d, per lb. 5s.

Dichloraniline. Per lb., 4s 6d.

Dinitrobenzene. 88/99°C., per lb., 2s 1d.

Dinitrotoluene. Drums extra. SP 15°C., per lb., 2s 1½d; SP 26°C., per lb., 1s 5d; SP 33°C., per lb., 1s 2½d; SP 66/68°C., per lb., 2s 1d.

p-Nitraniline. Per lb., 5s 1d.

Nitrobenzene. Spot, 90 gal. drums (drums extra), 1-ton lots d/d, per lb. 10d.

Nitronaphthalene. Per lb., 2s 5½d.

o-Toluidine. 8-10 cwt. drums (drums extra), per lb., 1s 11d.

p-Toluidine. In casks, per lb., 6s 1d.

Dimethylaniline. Drums extra, c.p., per lb., 3s 5d.

Chemicals in U.K.—East Germany Trade Plan

CHEMICALS are among the British goods to be exported to East Germany under the recently concluded trade arrangement. Chemicals and fertilisers will also be imported from East Germany.

No details have been released of the types of chemicals involved.

Plastics, iron and steel, scientific instruments and industrial plant and equipment are also among the British exports. East Germany will send Britain paraffin wax, scientific instruments and machinery among a wide range of goods.

Chemical Fellowships

Applications by advanced students of chemistry are invited by the trustees of the Ramsay Memorial Fellowships Trust at University College, London.

One of the fellowships will be limited to candidates educated in Glasgow, who can apply to be considered for either fellowship. The value of each fellowship will be £600 per annum, to which may be added a grant for expenses of research not exceeding £100 per annum. The fellowships will normally be tenable for two years.

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TRADE NOTES

Glycerine Price Raised

Prices of their glycerine, unchanged since a reduction of £47 10s. per ton in August 1956, have now had to be increased, state Glycerine Ltd., 8 Tudor Street, London E.C.4. The price of refined glycerine has been raised by £20 per ton from 26 January. A new price list is available from the company.

First MoS₂ Aerosol

Moly-Paul (K. S. Paul (Molybdenum Disulphide) Ltd., Great Western Trading Estate, Park Royal Road, London N.W.10), claim to have produced the first British-made MoS₂ aerosol container and the first to incorporate an agitator. Tests on selected applications have shown that Moly-vapour-bond is not only a convenient, easy and clean method of application, but that it is also a most effective solid lubricant film. It can be used as an anti-seize material for hot and cold screw threads, for dry lubrication for machinery and instruments, and for surface lubrication (operating temperature range is -128°C to +370°C).

Instruments Demonstrated

Among instruments manufactured by Beckman Instruments Inc. and demonstrated at the Abercorn Rooms, Gt. Eastern Hotel, London E.C.2, by A. Gallenkamp and Co. Ltd., Sun Street, London E.C.2, was the industrial gas chromatograph for monitoring four components in up to 10 streams on plant.

In the demonstration commercial propane was analysed, using a double column and hydrogen as the carrier gas. Also on show were an electrolytic hygrometer for monitoring stream humidity up to 1,000 p.p.m., a dissolved oxygen analyser with a sensitivity of 25 parts per thousand million for boiler water feed, a portable paramagnetic oxygen analyser and a laboratory gas chromatograph. After the demonstrations the instruments were taken to a number of industrial firms for plant trials.

Change of Address

From 26 January the London office of Sunvic Controls Ltd. will be at Crown House, Aldwych, London W.C.2. (Temple Bar 8040.)

Marketing Oil Pollution Chemical

Further to the announcement that C. H. Bailey and Co., Newport, had developed a chemical compound for separating the oil from the water in sludge left in oil tankers (CHEMICAL AGE, 24 January, p. 185) the company have formed Crow Oil (Sales) Ltd., Perch Buildings, 20 West Bute Street, Cardiff, to market the compound, which is described as a de-emulsifier.

Pressure Gauges

Ivix pressure gauges are described in a leaflet, G/46, by Negretti and Zambra, 122 Regent Street, London W.1. Features include accuracy $\frac{1}{2}\%$ or 1% according to

range, high overload and underload capacity, precision cut rack and pinion, zero adjustment, overload and underload stops fitted to movement, special diaphragm capsule movement for low ranges, specially designed Bourdon tube for high ranges.

To Distribute German Chemicals

Charles H. Windschuegl Ltd., 1 Leadenhall Street, London E.C.3, have been appointed sole distributors in the U.K. for a wide range of chemical products of Chemische Fabrik Stockhausen and Cie., of Krefeld. They include Antispumin HF, and Antispumin 7517, anti-foaming agents with uses in many industries.

Diversey (U.K.) and Deosan

Diversey (U.K.) Ltd., 42-46 Weymouth Street, London W.1, point out that their parent company, Diversey Corporation of Chicago, U.S., have acquired the factory of Deosan Ltd. of Riddings, Derbyshire. The Denver Manufacturing Co. (CHEMICAL AGE, 17 January p. 134) have not been involved in the recent take over and have no association whatsoever with Deosan Ltd.

Roller Coverings

A new booklet on roller coverings has been published by the Dunlop General Rubber Goods Division, Cambridge Street, Manchester 1. Properties and features of natural rubber, nitrile, butyl, neoprene, silicone rubber and p.t.f.e., are given. Also included are notes on the use and maintenance of rollers.

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NEW PATENTS

By permission of the Controller, HM Stationery Office, the following extracts are reproduced from the 'Official Journal (Patents)', which is available from the Patent Office (Sale Branch), 25 Southampton Buildings, Chancery Lane, London W.C.2, price 3s 6d including postage; annual subscription £8 2s.

Specifications filed in connection with the appearances in the following list will be open to public inspection on the dates shown. Opposition to the grant of a patent on any of the applications listed may be lodged by filing patents from 12 at any time within the prescribed period.

ACCEPTANCES

Open to public inspection 4 March

Polymeric isocyanates and their manufacture. Imperial Chemical Industries, Ltd., France, H. and Lister, A. 809 809

Modified formaldehyde polymers and method of preparing them. Du Pont De Nemours & Co., E. I. 809 754

Process for the working up of crude alkali metal terephthalates. Henkel & Cie. G.m.b.H. 809 755

Preparation of isopropyl benzenes. Bataafsche Petroleum Maatschappij N.V., De. 809 909

Process for compacting very finely particled solids with low bulk density. Badische Anilin- & Soda-Fabrik A.G. 809 756

Production of olefin polymers. Badische Anilin- & Soda-Fabrik A.G. 809 717

Manufacture of viscose structures. Glanzstoff-Courtaulds G.m.b.H. 809 720

Compositions for the control of parasites in animals. Dow Chemical Co. 809 814

Recovery of sucrose esters. Distillers Co. Ltd. 809 815

Manufacture of methyl sulphide and methyl mercaptan. Abbey, A. (Dow Chemical Co.). 810 017

Vacuum-tight metal to ceramic joints. Telefunken G.m.b.H. 809 915

Pharmacologically active piperazine derivatives and processes for preparing them. Laboratoria Pharmaceutica Dr. C. Janssen N.V. 809 760

Purification of cracking gases. Chemische Werke Hüls A.G. 809 888

Nitrile rubber compositions and accessories manufactured therefrom for electric cables. Pirelli S.p.A. 809 722

Recovery of indene by azeotropic purification with furfural. Neville Chemical Co. 809 992

Roasting zinc sulphide ores. New Jersey Zinc Co. 809 765

Method of removing scale from zirconium. Unternehmungen Der Eisen-Und Stahlindustrie A.G. Für. 809 892

Oxidation of alpha-tocopherol. Eastman Kodak Co. 809 895

Organohydrogenosiloxane emulsions and the treatment of fabrics therewith. Midland Silicones Ltd. 809 766

Method of preparing di-esters of phthalic acids. Imhausen Werke G.m.b.H. 809 730

Organosiloxane greases. Midland Silicones Ltd. 809 731

Manufacture of N-alkyl- and N-cycloalkylsulphur (2-benzothiazolesulphen) amides. United States Rubber Co. 809 732

Recovery of terephthalic acid. Henkel & Cie G.m.b.H. 809 767

Process for producing thermoplastic polycarbonates. Farbenfabriken Bayer A.G. 809 735

Esterification process. Esso Research & Engineering Co. 809 738

Filter for gases and liquids. Etablissements R. Schneider, and Poelman, A. J. J. 809 739

Production of cyclo-octane. Badische Anilin- & Soda-Fabrik A.G. 809 740

Process for the preparation of aromatic monocarboxylic acids. Mid-Century Corp. [Divided out of 807 091.] 810 020

Steroids and the manufacture thereof. Upjohn Co. [Divided out of 809 046.] 809 820

Open to public inspection 11 March

Production of uranium. Spalding, F. H., Wilhelm, H. A., and Keller, W. H. 810 033

Sequestration of ferric iron in caustic alkali solutions. Evans & Rais, Ltd., N. 810 113

Refining metal by electrode-deposition. U.K. Atomic Energy Authority. 810 091

Treatment of gases with liquids. Whessoe, Ltd. 810 192

Cephalosporin C. National Research Development Corp. [Cognate application 17 379.] 810 196

Process for forming granules from powdered materials and apparatus for use in such process. Fisons, Ltd. 810 290

Production of oil gas of low cost gas of desired specification. Stark, V. 810 094

Polymerisation of olefins and the linear high molecular weight polymers produced thereby. Montecatini Soc. Generale per l'Industria Mineraria e Chimica, and Ziegler, K. 810 023

Method of extracting and purifying a pancreatic hormone acting on the respiratory centres. Santenoise, J. P. D. 810 204

Foamed polyester resin laminated products. Hudson Foam Plastics Corp. 810 095

Stilrene monoazodestuffs and process for their manufacture. Ciba Ltd. 810 297

Hydrocarbon oil compositions and additives therefor. Bataafsche Petroleum Maatschappij N.V., De. 810 206

Metaliferous complexes of mono-azo dyestuffs of the acylacetic amide series, and their preparation. Compagnie Francaise des Matières Colorantes. 810 207

Apparatus for the production of alkali cellulose. Courtaulds, Ltd. 810 300

Fertilisers. Fisons, Ltd. [Cognate application 3 555.] 810 208

Production of insulative coatings on steel strip. Armcro International Corporation. 810 039

Amines. May & Baker, Ltd. [Addition to 769 706.] 810 304

Hydrocarbon polymers and the preparation thereof. Du Pont de Nemours & Co., E. I. 810 210

Acid esters of starch. Upjohn Co. 810 306

Process for dressing artificial filaments and fibres to give a soft feel. Spinnfaser, A.G. 810 040

Method for the preparation of aminoacyl anilides. Astra Apothekernes Kemiske Fabriker A.B. 810 212

Amino acetic acids and their use. Geiger, A.G., J. R. 810 126

Process of curing siloxane resins. Midland Silicones, Ltd. 810 283

Polymeric materials. Imperial Chemical Industries, Ltd. 810 125

Chloromethylation process. Imperial Chemical Industries, Ltd. 810 026

Water-repellent materials. General Electric Co. 810 310

Bleaching compositions. Whiffen & Sons, Ltd. 810 128

Fungicides. Farbenfabriken Bayer A.G. 810 044

Resins. Midland Silicones, Ltd. 810 284

Phenylcarbamates and their preparation. Laboratoires Dausse. 810 313

Treatment of Raney metal suspensions. Celanese Corp. of America. 810 263

Manufacture of a 17-hydroxysteroid. Farbwerke Hoechst A.G. 810 264

Anion-exchange resins. Permutit Co., Ltd. 810 027

Process for the production of gas containing hydrogen by the conversion of hydrocarbons. Office National Industriel de l'Azote. [Addition to 772,787.] 810 265

Methods for controlling the degree of polymerisation in the polymerisation or copolymerisation of ethylenically unsaturated monomers. Du Pont de Nemours & Co., E. I. 810 268

Drier units applicable to refrigerating systems. Ansol Chemical Co. 810 216

Aspirin crystallisation. Monsanto Chemicals, Ltd. 810 050

Method of coating metals. Chromalloy Corp. 810 051

Method of sealing glass to other elements. Pittsburgh Plate Glass Co. 810 322

Aluminium alcoholate derivatives. Hardman & Holden, Ltd., Kemp, S. G., and Bashford, V. G. 810 323

Stabilised polymerisable materials and process for preparation. Rohm & Haas Co. 810 221

Hardenable synthetic resinous compositions. Farbenfabriken Bayer A.G. 810 222

Production of polymerisation products from olefinically unsaturated hydrocarbons. Badische Anilin- & Soda-Fabrik A.G. 810 228

Minimising the electrostatic charge of undyed fibres, threads or fabrics. Badische Anilin- & Soda-Fabrik A.G. 810 352

Epoxide resins. Chemische Werke Albert. 810 230

Methods of sealing anodic aluminium oxide coatings. Aluminium Labs., Ltd. 810 231

Phthalazine compounds basically substituted in the 1-position. Cassella Farbwerke Mainkur A.G. 810 108

Sultams. Farbenfabriken Bayer A.G. 810 356

Preparation of worts. Brewing Patents, Ltd., Cook, A. H., Pollock, J. R. A., and Davis, A. D. 810 146

Dinitration of O-alkylphenols. Rohm & Haas Co. 810 239

Dehydrogenation of hydrocarbons. California Research Corp. 810 149

Detergent compositions. Henkel-Helios A. B. 810 151

Esters of aminoacids. Imperial Chemical Industries, Ltd. 810 332

Disazo dyestuffs derived from sulphonates. Farbenfabriken Bayer A.G. 810 246

Water-insoluble diphenylamine dyestuffs. Sandoz Ltd. 810 360

Liquid shortening. Hedley & Co., Ltd. T. 810 277

Stable pourable oleaginous suspensions. Hedley & Co., Ltd. T. 810 278

Purification of refractory metals. Du Pont de Nemours & Co., E. I. 810 086

Alkyl arylstearates and process of producing same. Institut Francais du Petrole, des Carburants et Lubrifiants. 810 087

Nickel-molybdenum-iron alloys. Mond Nickel Co., Ltd. 810 089

Apparatus and process for continuous ion-exchange. Infilco, Inc. 810 090

Preparation of chlorinated aryl oxybutyric acids. Monsanto Chemicals, Ltd. 810 154

Condensation of zinc from metallic vapours. National Smelting Co., Ltd. 810 158

Production of hydroxyethylated phenols. Badische Anilin- & Soda-Fabrik A.G. 810 069

Process for the preparation of lactams. Deutsche Gold- und Silber-Scheideanstalt Vorm. Roessler. 810 165

Polymerisation of isolefins. Esso Research & Engineering Co. 810 071

Coating process for polyethylene and composite articles thereby obtained. Dow Chemical Co. 810 169

Preparation of methylacrylamide. American Cyanamid Co. 810 170

Blood glue and process for making same. American-Marietta Co. 810 338

Manufacture of margarine. Knollenberg, R., Hahn, C. G., and Holdt, K. Von [trading as Schroeder & Co.]. 810 275



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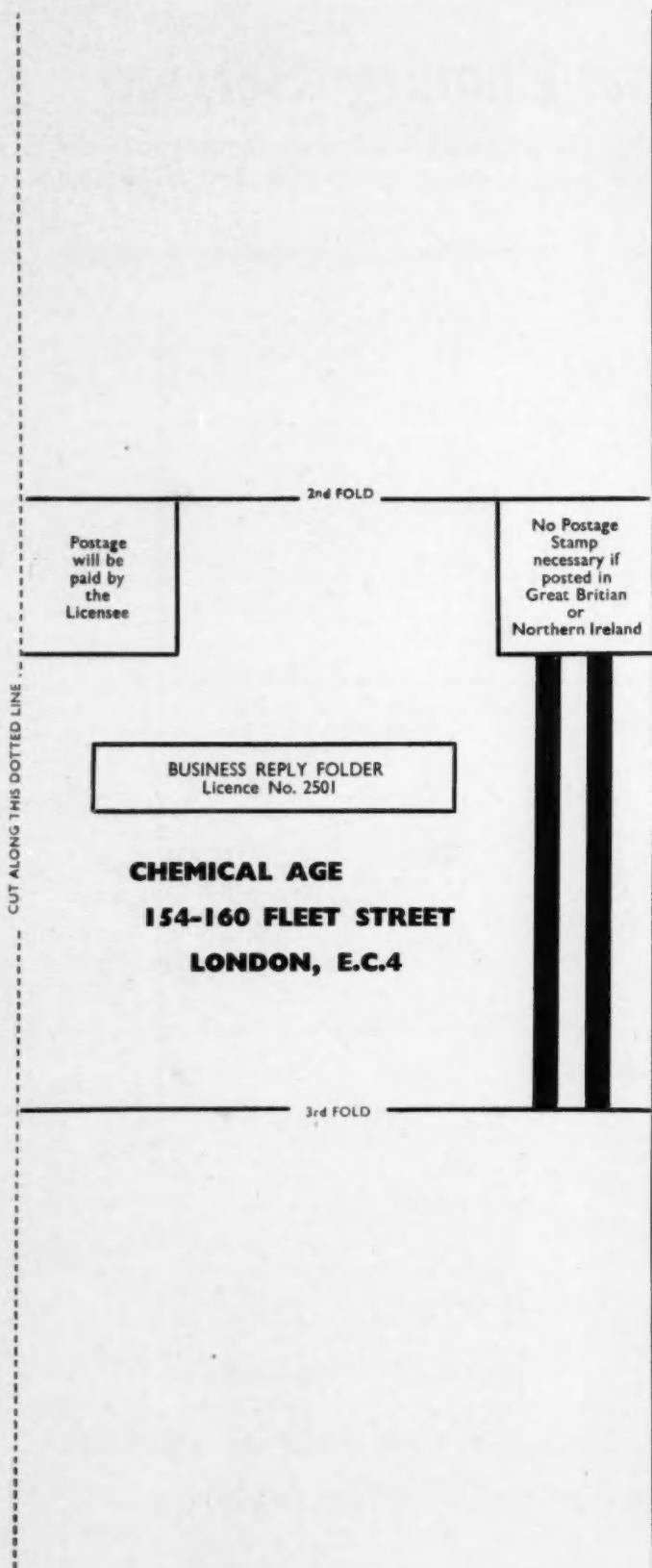
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